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Final
Sampling and Analysis Plan

Thames River
Pre-Design Sediment Sampling
at Inner Pier 1

for

Naval Submarine Base
New London

Groton, Connecticut

Naval Facilities Engineering Command
Mid-Atlantic

Contract Number N62470-08-D-1001

Contract Task Order WE24

September 2009

SAP Worksheet #1 – Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
June 16, 2009

UFP-SAP for Thames River Pre-Design Sediment Sampling at Inner Pier 1
Naval Submarine Base - New London
Groton, Connecticut

Prepared for:
Naval Facilities Engineering Command
Mid-Atlantic
9742 Maryland Avenue
Norfolk, Virginia 23511-3095

Prepared by:
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234 Mall Boulevard, Suite 260
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Prepared under:
Contract No. N62470-08-D-1001
Contract Task Order WE24

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Corey A. Rich

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NAVFAC Remedial Project Manager
Jim Gravette

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EPA Region 1 Remedial Project Manager
Kymberlee Keckler

Approval Signature: 9/9/09
CTDEP Environmental Analyst 3
Mark Lewis

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

DRAFT
SAMPLING AND ANALYSIS PLAN
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Tom Johnston

Approval Signature: kenneth.a.bowers Digitally signed by kenneth.a.bowers DN: cn=kenneth.a.bowers Reason: I have reviewed this document Date: 2009.06.26 15:53:12 -04'00'
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EPA Region 1 Remedial Project Manager
Kymberlee Keckler

Approval Signature:
CTDEP Environmental Analyst 3
Mark Lewis

EXECUTIVE SUMMARY

This Uniform Federal Policy (UFP)-Sampling and Analysis Plan (SAP) describes the investigation designed to collect additional sediment samples from the Thames River at Inner Pier 1 at Naval Submarine Base – New London located in Groton, Connecticut. The UFP-SAP was prepared by Tetra Tech NUS, Inc. (Tetra Tech) on behalf of Naval Facilities Engineering Command Mid-Atlantic under Contract Number N62467-08-D-1001, Contract Task Order WE24. The field investigation described herein will be conducted to collect additional sediment data that will be used to evaluate disposal options for the contaminated sediment. The data will be used in two documents including the Inner Pier 1 Engineering Evaluation/Cost Analysis (EE/CA) and remedial design work plan for the Non-Time-Critical Removal Action (NTCRA).

Sampling of sediment has already been conducted at Inner Pier 1 during previous investigations; however, sufficient data have not been collected to fully characterize the sediment for disposal. Remedial alternatives that include various disposal options are being evaluated for contaminated sediment. It is likely that the contaminated sediment will be removed and will require disposal at an appropriate upland facility (solid waste or hazardous waste) depending on contaminant concentrations in the sediment.

The environmental question being addressed by this UFP-SAP is the following:

- Are in-situ contaminant concentrations in the sediment proposed for removal under the NTCRA suitable for placement in a solid waste or hazardous/Toxic Substances Control Act (TSCA) waste landfill?

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Appendix E Meeting Minutes for March 16, 2009 and May 14, 2009 Partnering Meeting and June 17, 2009 Conference Call
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ACRONYMS

BERA	Baseline Ecological Risk Assessment
bss	below sediment surface
CA	Corrective Action
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CIF	Controlled Industrial Facility
CLEAN	Comprehensive Long-Term Environmental Action Navy
CSM	Conceptual Site Model
CTDEP	Connecticut Department of Environmental Protection
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Adsorption
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
EE/CA	Engineering Evaluation/Cost Analysis
EPA	Environmental Protection Agency
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPS	Global Positioning System
HASP	Health and Safety Plan
HSM	Health and Safety Manager
HSO	Health and Safety Officer
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
IDL	Instrument Detection Limit
IRCDQM	Installation Restoration Chemical Data Quality Manual
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management Systems
MDL	Method Detection Limit
MPC	Measurement Performance Criterion

MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	mean sea level
NAD	North American Datum
NAVFAC	Naval Facilities Engineering Command
NFESC	Naval Facilities Engineering Service Center
NIST	National Institute of Standards and Technology
NOAA	National Oceanic and Atmospheric Administration
NSB-NLON	Naval Submarine Base – New London
NTCRA	Non-Time-Critical Removal Action
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PAH	Polynuclear Aromatic Hydrocarbon
PAL	Project Action Limit
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB	Polychlorinated Biphenyl
PHSO	Project Health and Safety Officer
PM	Project Manager
POC	Point of Contact
PRG	Preliminary Remediation Goal
PT	Proficiency Testing [previously known as performance evaluation (PE) sample]
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
%R	Percent Recovery
RCRA	Resource Conservation and Recovery Act
RCSA	Regulations of Connecticut State Agencies
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SQLs	Sample Quantitation Limits or Structured Query Language

SSO	Site Safety Officer
SVOA	Semivolatile Organic Analyte
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TSCA	Toxic Substances Control Act
UFP	Uniform Federal Policy
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compound
WCSD	Watershed Contaminated Source Document

SAP Worksheet #2 -- SAP Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Inner Pier 1
Operable Unit: Inner Pier 1 is a portion of OU4
Contractor Name: Tetra Tech NUS, Inc.
Contract Number: N62470-08-D-1001
Contract Title: Navy CLEAN
Work Assignment Number (optional): CTO WE24

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP) (EPA, 2005) and EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (2002). Identify any additional guidance used to prepare SAP: None

2. Identify regulatory program: CERCLA for site investigation, RCRA for evaluation of disposal options.

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Partnering Meeting conducted with Navy, EPA, CTDEP, Tetra Tech NUS, Inc., and Tetra Tech ECI – Scope of sampling effort discussed by team.	March 16, 2009

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Final Thames River Validation Study Work Plan, Sampling and Analysis Plan, and Screening Level Environmental Risk Assessment by Battelle and Neptune and Company	October 2004
Addendum 1, Thames River Validation Study Work Plan by Battelle and Neptune and Company	February 2007
Final Thames River Validation Study by Battelle and Neptune and Company	March 2008a
Final Engineering Evaluation/Cost Analysis for Pier 1 Inner Area by Battelle	March 2008b
Sediment Sampling and Analysis Plan, Waterfront Maintenance Dredging, Naval Submarine Base New London by TEC Inc.	April 2008
Lower Subase Remedial Investigation Report by Tetra Tech NUS, Inc.	January 1999
Draft Lower Subase Feasibility Study Report by Tetra Tech NUS, Inc.	March 2008
Final Sampling and Analysis Plan for Thames River Sediment Sampling at Zone 4, Pier 1, and Outer Pier 1 by Tetra Tech NUS, Inc.	October 2008

6. List organizational partners (stakeholders) and connection with lead organization:
EPA (regulatory oversight), CTDEP (regulatory oversight), NOAA (regulatory oversight), and NAVFAC Mid-Atlantic (property owner).

7. Lead organization (see Worksheet #7 for detailed list of data users) NAVFAC Mid-Atlantic

- If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Cross-walk omitted, not needed.

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address or Mailing Address	Document Control Number (Optional)
James Gravette	NAVFAC Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	757-444-0735	james.gravette@navy.mil	NA
Richard Conant	Environmental Restoration Program Manager	NSB-NLON	860-694-5649	richard.conant@navy.mil	NA
Bonnie Capito	Administrative Record/Librarian	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	NA
Kymberlee Keckler	RPM	EPA Region 1	617-918-1385	Keckler.Kymberlee@epamail.epa.gov	NA
Mark Lewis	Environmental Analyst 3	CTDEP	860-424-3768	mark.lewis@po.state.ct.us	NA
Ken Finkelstein, Ph.D.	Environmental Scientist	NOAA	671-918-1499	Ken.Finkelstein@NOAA.gov	NA
Corey Rich	Base Coordinator/ Project Manager (PM)	Tetra Tech	412-921-8984	corey.rich@tetratech.com	NA
Aaron Bernhardt	Ecological Risk Assessor	Tetra Tech	412-921-8433	aaron.bernhardt@tetratech.com	NA
Nina Balsamo	Project Engineer	Tetra Tech	412-921-8132	nina.balsamo@tetratech.com	NA
Joe Samchuck	Data Validation Manager	Tetra Tech	412-921-8510	joseph.samchuck@tetratech.com	NA
TBD	Field Operations Leader (FOL)/Site Safety Officer(SSO)	Tetra Tech	TBD	TBD	NA

Name of SAP Recipient	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address or Mailing Address	Document Control Number (Optional)
Leanne Ganser	Project Chemist	Tetra Tech	412-921-8148	leanne.ganser@tetratech.com	NA
Todd Finlayson	EPA Contractor	Gannett Fleming	TBD	rtfinlayson@gfnet.com	NA
Ed Lawler	Analytical Laboratory Project Manager	Mitkem	401-732-3400	elawler@mitkem.com	NA

Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of this SAP to all project personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet)

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and insure that the completed sign-off sheet is included in the central project file.

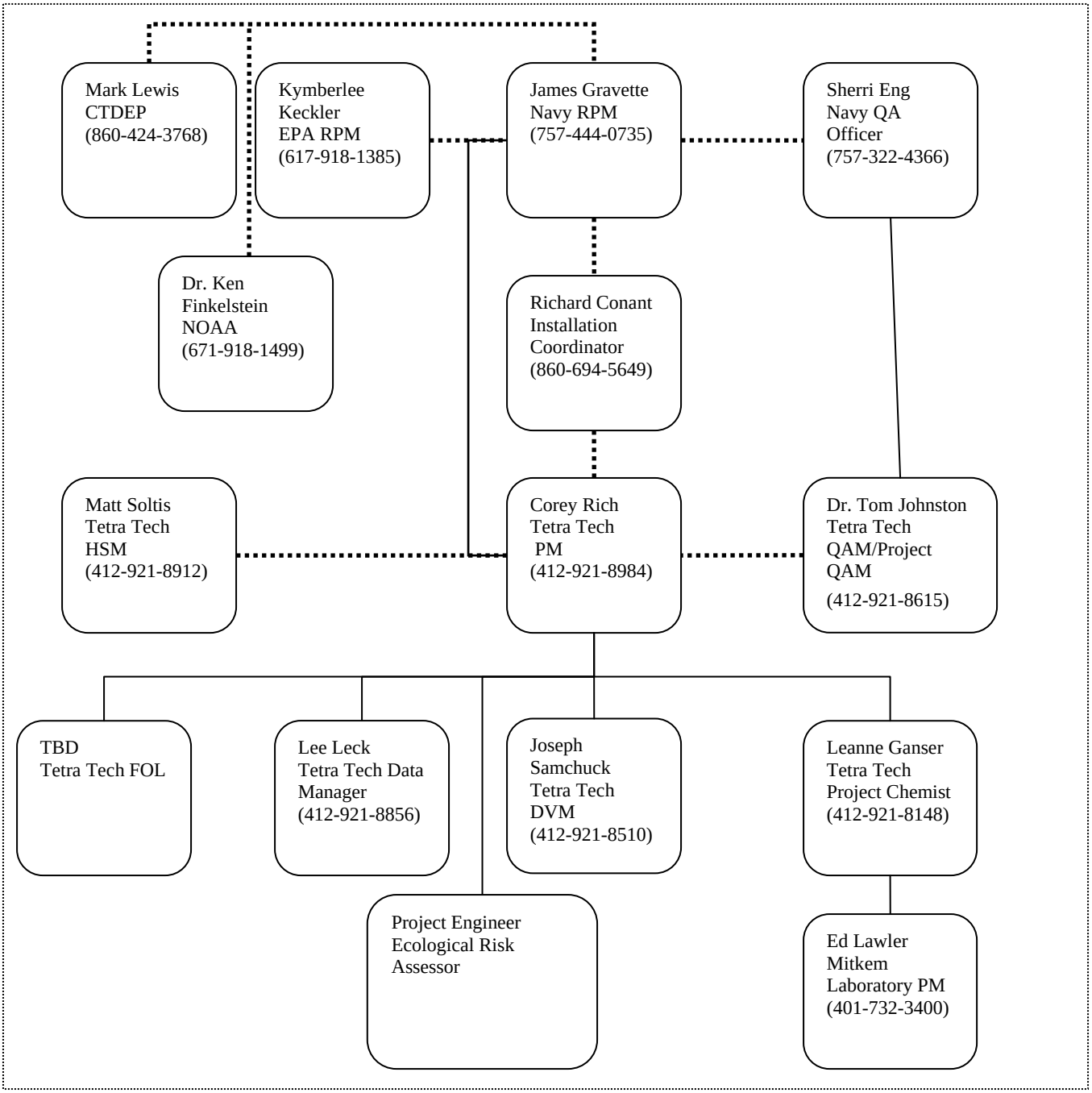
Name	Organization/Title/Role	Telephone Number (optional)	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Corey Rich	Tetra Tech/PM	412-921-8984		All	
TBD	Tetra Tech/FOL and SSO	TBD		All	
TBD	Tetra Tech/Project Health and Safety Officer (PHSO)	TBD		Worksheets #10, #11, #14 and Health and Safety Plan (HASP)	
Leanne Ganser	Tetra Tech/Project Chemist	412-921-8148		All	
Dr. Tom Johnston	Tetra Tech/Quality Assurance Manager (QAM)	412-921-8615		All	
Matt Soltis	Tetra Tech/Health and Safety Manager (HSM)	412-921-8912		HASP	
Joseph Samchuck	Tetra Tech/Data Validation Manager (DVM)	412-921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	
Lee Leck	Tetra Tech/Data Manager	412-921-8856		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	
Ed Lawler	Mitkem Laboratory PM	401-732-3400		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	

Name	Organization/Title/Role	Telephone Number (optional)	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Aaron Bernhardt	Tetra Tech/Ecological Risk Assessor	412-921-8433		TBD	
Nina Balsamo	Tetra Tech/Project Engineer	412-921-8132		TBD	

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ————
Lines of Communication ·····



SAP Worksheet #6 -- Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
QAPP amendments	Tetra Tech PM NAVFAC RPM	Corey Rich James Gravette	412-921-8984 757-444-0735	After realizing an amendment is needed, PM will send NAVFAC a concurrence letter within 7 days. The NAVFAC RPM will sign the letter within 5 days of receipt.
Schedule changes	Tetra Tech PM	Corey Rich	412-921-8984	When impact is realized, PM will send NAVFAC a schedule concurrence letter within 7 days or prior to the first affected deliverable date.
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL NAVFAC RPM Tetra Tech PM	TBD James Gravette Corey Rich	TBD 757-444-0735 412-921-8984	FOL informs PM the day the issue is realized. PM informs the NAVFAC RPM of the issue within 1 day of the FOL's notification. PM also sends a concurrence letter to the NAVFAC within 7 days if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on a Field Task Modification Request (FTMR) form.
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM	TBD Corey Rich Dr. Tom Johnston	TBD 412-921-8984 412-921-8615	On the day the problem is identified, FOL, SSO, or QAM informs PM and documents problem on a FTMR. On the same day of the notification from the FOL, PM informs NAVFAC RPM and Installation coordinator.
Field or laboratory data issues	Laboratory PM Tetra Tech Project Chemist	Ed Lawler Leanne Ganser	401-732-3400 412-921-8148	When issue is related to chemical data, Laboratory PM notifies Project Chemist within 2 days of identification of problem. Project Chemist notifies Tetra Tech PM within 1 day.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Corey Rich	PM	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project. Specific responsibilities: • Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations. • Functions as the primary Tetra Tech interface with the NAVFAC RPM, regulators, Tetra Tech field and office personnel, and laboratory points of contact. • Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories. • Monitors and evaluates all Tetra Tech subcontractor performance. • Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data interpretation and report preparation). • Coordinates and oversees maintenance of all Tetra Tech project records. • Coordinates and oversees review of Tetra Tech project deliverables. • Prepares and issues final Tetra Tech deliverables to NAVFAC.	B.A., Physics, B.S., Civil Engineering, 18 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
TBD	FOL	Tetra Tech	Supervises, coordinates, and performs field sampling activities. Specific responsibilities: • Functions as the on-site communications link between field staff members, subcontractors, and Tetra Tech PM. • Oversees mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to work schedules provided by the Tetra Tech PM. • Ensures proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs (field change orders) when necessary. • Identifies and resolves problems in the field, implements and documents corrective action (CA) procedures, and provides communication between the field team and project management. Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • Ensures that all health and safety requirements for the investigation are implemented.	TBD

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
			• Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • FOL responsibilities include initiating and conducting equipment inventories to ensure equipment is available, purchasing equipment as required, staging equipment for efficient loading and transport from the Tetra Tech office to the site, and, after field activities are completed, demobilizing the equipment.	
TBD	SSO	Tetra Tech	The SSO will be responsible for training and monitoring site conditions. Details of these responsibilities are presented in the HASP and include: • Controlling specific health and safety-related field operations such as personnel decontamination, monitoring of worker heat or cold stress, and distribution of safety equipment. • Conducting and documenting a daily health and safety briefing each day while on site. • Assuring that field personnel comply with all procedures established in the HASP. • Identifying SSOs in their absence. • Terminating work if an imminent safety hazard, emergency situation, or other potentially dangerous situation is encountered.	TBD

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
			• Ensuring the availability and condition of health and safety monitoring equipment. • Coordinating with the FOL and Tetra Tech PM to institute and document any necessary HASP modifications. • Ensuring that facility personnel and subcontractors are adequately advised and kept clear of potentially contaminated materials.	
Dr. Tom Johnston	QAM	Tetra Tech	Reviews the SAP, oversees preparation of lab scope, and conducts data quality reviews. Ensures that quality aspects of the CLEAN program are implemented. Specific responsibilities: • Develops, maintains, and monitors QA policies and procedures. • Provides training to Tetra Tech staff in QA/quality control (QC) policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, SAP requirements, and corporate policies and procedures. • Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of CA plans and ensuring correction of non-conformances reported in internal or external audits.	Ph.D. Analytical Chemistry, 32 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
			• Ensures that this SAP meets Tetra Tech, Navy, and EPA QA requirements. • Prepares QA reports for management.	
Joseph Samchuck	DVM	Tetra Tech	Manages data validation activities within Tetra Tech, including: • Ensuring QA of data validation deliverables. • Providing technical advice on data usability. • Coordinating and maintain data validation review schedule.	MBA, M.S. Finance, B.S. Chemistry, 24 years environmental experience
Matt Soltis	HSM	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety Program including: • Providing technical advice to the Tetra Tech PM on matters of health and safety. • Overseeing development and review of the HASP. • Conducting health and safety audits. • Preparing health and safety reports for management.	CIH, CSP, B.S. Industrial Safety Sciences, 24 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Leanne Ganser	Project Chemist	Tetra Tech	Provides support to the project including: • Assisting in preparation of SAP. • Preparing laboratory scopes of work, coordinates analyses with laboratory chemists, ensures that the laboratory scope of work is followed, and communicates with Tetra Tech staff. • Providing technical advice to the Tetra Tech team on matters of project chemistry. • Reviewing laboratory data. • Evaluating data usability. • Functioning as the primary interface between the subcontracted laboratory and Tetra Tech staff.	M.S., Environmental Science, 4 years environmental experience
Ed Lawler	Laboratory PM	Mitkem	Coordinates analyses with lab chemists, ensures that the scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.	Can be provided upon request

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA), 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the corporate Tetra Tech HASP.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Additional Sediment Sampling at Inner Pier 1 Projected Date(s) of Sampling: September 2009 Project Manager: Corey Rich	Site Name: Thames River at NSB-NLON Site Location: Groton, Connecticut

Date of Session: March 16, 2009
Scoping Session Purpose: Determine Scope and DQOs of Project

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Corey Rich	Project Manager	Tetra Tech NUS, Inc.	412-921-8984	corey.rich@tetratech.com	Project Management
Val Jurka	Acting RPM	Navy	757-322-8319	val.jurka@navy.mil	RPM
Richard Conant	Environmental Restoration Program Manager	Navy	860-694-5649	richard.conant@navy.mil	Base Point of Contact
Kymberlee Keckler	RPM	EPA	617-918-1385	Keckler.Kymberlee@epamail.epa.gov	RPM
Mark Lewis	Environmental Analyst 3	CTDEP	860-424-3768	mark.lewis@po.state.ct.us	Regulatory Review
Dan Sullivan	Project Manager	Tetra Tech ECI	NA	dan.sullivan.tteci.com	Project Manager for Remediation Work
Bart Hoskins	Ecological Risk Assessor	EPA	NA	Hoskins.Bart@epamail.epa.gov	Regulatory Review
Ken Finklestein	Environmental Scientist	NOAA	671-918-1499	Ken.Finkelstein@NOAA.gov	Regulatory Review
Todd Finlayson	Ecological Risk Assessor	Gannett Fleming	NA	rtfinlayson@gfnet.com	Regulatory Review
Aaron Bernhardt	Ecological Risk Assessor	Tetra Tech	412-921-8433	aaron.bernhardt@tetratech.com	Ecological Risk Assessment
Nina Balsamo	Civil Engineer	Tetra Tech	412-921-8132	nina.balsamo@tetratech.com	Project Engineer

Decisions on the additional sediment sampling at Inner Pier 1 were captured in the Minutes from the March 16, 2009 and May 14, 2009 Partnering Meetings and June 17, 2009 Conference Call which are provided in Appendix E.

SAP Worksheet #10 -- Problem Definition
(UFP-QAPP Manual Section 2.5.2)

10.1 SITE DESCRIPTION

10.1.1 Facility Background

NSB-NLON is located in southeastern Connecticut in the Towns of Ledyard and Groton and is situated on the eastern bank of the Thames River, approximately 6 miles north of Long Island Sound (Figure 10-1). The Navy's use of the Thames River shoreline at NSB-NLON began in 1868, but most of the construction along the river at NSB-NLON took place in the early 1900s, with a major expansion from 1935 to 1940. NSB-NLON currently provides base command for Naval submarine activities in the Atlantic Ocean. It also provides housing for Navy personnel and their families and supports submarine training facilities, military offices, medical facilities, and facilities for submarine maintenance, repair, and overhaul.

The Lower Subase contains piers and berths for submarine docking; facilities for submarine maintenance, repair, and overhaul; and administrative buildings (Figure 10-1). Battery overhaul was one of the largest operations at the Lower Subase prior to the advent of nuclear-powered submarines. Lead-acid battery maintenance and overhaul activities were conducted in this area until the mid-1950s. A classified materials incinerator was also operated in the Lower Subase until 1967. It is possible that the resulting ash was disposed in portions of the Lower Subase. Petroleum products were used by the Navy throughout the Lower Subase. Releases of petroleum products to the environment may have occurred because of leaks from subsurface storage tanks and fuel distribution lines, vehicle and locomotive maintenance operations and associated waste disposal practices, and marine fueling activities. Other ship and submarine maintenance activities (e.g., painting) were also conducted in the Lower Subase and adjacent Thames River.

10.1.2 Thames River

The lower portion of the Thames River is highly industrialized with numerous water-dependent industries (pharmaceutical, oil, chemical, and manufacturing), power plants, and waste treatment plants located along its banks. Non-point discharges from road, agricultural, and other runoff sources occur throughout the river's drainage basin. The Thames River and its tributaries drain approximately 1,471 square miles of eastern Connecticut, western Rhode Island, and south-central Massachusetts. The Thames River is a salt wedge estuary that is highly stratified, with fresher water on the surface and denser saline water beneath. The average freshwater flow discharging to Long Island Sound from the Thames River has been estimated at 222 million cubic feet per day (Soderberg and Bruno, 1971); however, freshwater flow in the Thames River is small in comparison to intertidal volume and exchange (Bohlen and Tramontano, 1977). The 7-day, 10-year low-flow rate for the freshwater portion of the Thames River at NSB-NLON is estimated to be 146.3 cubic feet per second. A dredged navigational channel runs north to south in the Thames River. At NSB-NLON, the dredged channel depth is approximately 40 feet below mean sea level

(msl); depths outside the channel are shallow (2 to 10 feet), and the width of the river varies from approximately 600 to 900 feet.

The Navy conducts maintenance dredging in the Thames River adjacent NSB-NLON to maintain water depths required for the submarine fleet. The most recent dredging projects were conducted in 1995/1996 and 2006. Dredged material from the projects was disposed at a designated open-water disposal site in Long Island Sound and in a Confined Aquatic Disposal (CAD) cell constructed in the Thames River just downstream of NSB-NLON. Plans for additional maintenance dredging in numerous other areas along the Lower Subase are currently being prepared by the Navy.

CTDEP has classified Thames River water quality as SC/SB, which designates the water for marine fish, shellfish, and wildlife habitat, certain aquaculture operations, recreational uses, and industrial and other legitimate use, but the waters presently are not meeting water quality criteria or are not supporting one or more designated uses as a result of point or non-point sources of pollution (CTDEP, 2002). Sources contributing to this impairment include marinas, waterfowl, urban runoff/storm sewers, industrial and municipal point source discharges, and sanitary sewer overflows.

The Thames River adjacent to NSB-NLON was investigated during several phases, and a Watershed Contaminated Source Document (WCSD) was prepared by the Navy in 2007 for the Lower Portion of the Thames River. The results of these investigations showed that Navy sources (e.g., Marine Railway) contributed to contamination found in Thames River sediment in proximity to NSB-NLON. The Navy has implemented measures (e.g., time-critical removal actions) that will minimize future contaminant releases to the Thames River.

10.1.3 Inner and Outer Pier 1

The former Marine Railway at Pier 1 was identified as a source of contamination to the Thames River in 1998 during construction of the Controlled Industrial Facility (CIF). The former Marine Railway was operated at Pier 1 from approximately 1930 to 1960 and was used to pull ships out of the water for sandblasting/paint scraping and maintenance. Paint scraping activities often took place on the apron of the pull-out area. Pre-construction surveys for the CIF found elevated Polynuclear Aromatic Hydrocarbon (PAH) concentrations in subsurface soil and high concentrations of lead extending to bedrock. Sediment sampling identified high levels of PAHs and PCBs. The Pier 1 site was divided into two subareas (Inner and Outer) based on the distribution of contamination (see Figure 10-2).

Inner Pier 1 is approximately 190 feet long and 80 feet wide, with the western boundary being the northern portion of Pier 1. This portion of Pier 1 is constructed on a solid concrete foundation and extends approximately 175 feet from the CIF building (Building 476) into the Thames River. The remaining, pile-supported portion of Pier 1 is currently being demolished by the Navy and will be completely removed by July 2009. Boats formerly docked at Pier 1 have been moved out of the area by

the Navy to a new location. The sediment in the Thames River directly beneath Pier 1 has not been investigated. All Inner Pier 1 sediment and a majority of the contaminated Outer Pier 1 sediment will be remediated through a Non-Time-Critical Removal Action (NTCRA). The contaminated sediment remaining in Outer Pier 1 after the NTCRA will be addressed under a different remedial strategy determined through the Lower Subase Feasibility Study and subsequent proposed Plan and Record of Decision.

Elevated concentrations of contaminants were found in Inner Pier 1 sediment; therefore, this site is being addressed by the Navy through an Engineering Evaluation/Cost Analysis (EE/CA) (Battelle, 2008b) and NTCRA. The Navy plans to include portions of Outer Pier 1 with the Inner Pier 1 NTCRA to expedite remediation of the area and minimize remediation costs. The Navy intends to document this decision process in the Action Memorandum for the NTCRA. The remaining portions of Outer Pier 1 will be addressed through the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Remedial Investigation (RI)/Feasibility Study (FS) process because the contaminants and concentrations were similar to those found in Thames River sediment along Zone 4, another area of investigation to the north of Pier 1. Numerous sediment samples (Figure 10-2) were previously collected from the Inner Pier 1 area; however, additional sampling is needed to complete initial characterization of the sediment for waste disposal.

Outer Pier 1 is approximately 330 feet long and 150 feet wide, with the eastern boundary being the Thames River shoreline and the western boundary being the former Pier 1 (Figure 10-2). Outer Pier 1 was included in the Thames River Validation Study (Battelle, 2008a) and Baseline Ecological Risk Assessment (BERA) that were conducted to verify the results of the Screening Level Ecological Risk Assessment (SLERA) and to develop ecological cleanup goals. The conclusion of the Thames River Validation Study BERA was that the sediment of Outer Pier 1 was a medium of concern that required further evaluation. Numerous samples (Figure 10-2) were previously collected from the Outer Pier 1 area to complete delineation of contamination.

10.2 CONCEPTUAL SITE MODEL

The ecological conceptual site model (CSM) is shown on Figure 10-3. Sources of contamination and release mechanisms were discussed in Sections 10.1.3 for the study area.

Surface water transport is considered the primary transport mechanism for contamination in the Thames River at Inner Pier 1. Surface water transport occurs from both direct discharges to the river and from indirect discharges from the storm sewer system at outfalls. Deposition of particles from ship maintenance activities was a historical transport pathway.

10.3 PROBLEM DEFINITION

Sampling of sediment has already been conducted at Inner and Outer Pier 1 during previous investigations, and the extent of contamination, as defined by previously established ecological Preliminary Remediation Goals (PRGs), has been determined. However, remedial alternatives that include various disposal options are being evaluated in the EE/CA for the contaminated sediment. It is anticipated that the contaminated sediment will be removed and will require disposal at an upland facility. The type of upland facility [solid waste or hazardous/Toxic Substances Control Act (TSCA) waste] selected for disposal of the sediments will depend on contaminant concentrations and their characteristics in the sediment. Sediment samples will be tested for TCLP metals to determine if it is a characteristic hazardous waste and sediment samples will be tested for PCB Aroclors to determine if it is a TSCA waste. The analytical program for specific sample intervals will be determined by the availability of historical data. Some areas have historical PCB Aroclor data, and new samples from those areas will not be re-analyzed for PCB Aroclors. A representative number of samples will be analyzed for TCLP metals.

The environmental question to be addressed by this project is the following:

- Are in-situ contaminant concentrations in the sediment proposed for removal under the NTCRA suitable for placement in a solid waste or hazardous/TSCA waste landfill?

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

11.1 IDENTIFICATION OF STUDY GOALS

Decision Statement #1: Determine whether in-situ contaminant concentrations in Inner Pier 1 sediment meet the disposal requirements for a solid waste or hazardous/TSCA waste landfill. If the sediment meets the requirements of a solid waste landfill, the contaminated sediment can be disposed in this type of landfill; otherwise, the sediment will need to be further characterized ex-situ and then treated and/or disposed in a hazardous/TSCA waste landfill. For the PCB-contaminated sediment, it will be disposed based on its in-situ concentrations.

11.2 INFORMATION INPUTS

Sediment data collected from previous investigations [Marine Railway Investigation (SAIC, 1999), Final Thames River Rapid Sediment Characterization Pilot Study Survey Report (Battelle, 2003), and Thames River Validation Study (Battelle, 2004)] will be used in conjunction with newly collected sediment data to define the in-situ characteristics of the Inner Pier 1 sediment to initially identify the appropriate waste disposal option. Based on the results of the previous investigations, the new samples will be analyzed for a focused list of chemicals, including PCBs and Toxicity Characteristic Leaching Procedure (TCLP) metals. Samples will be analyzed using methods (SW-846 8082 and SW-846 1311/6010/7470) that can attain Quantitation Limits (QLs) less than the limits used to identify TSCA waste per federal (40 CFR 761) regulations and characteristic hazardous waste per federal (40 CFR 261) and Connecticut [Regulations of Connecticut State Agencies (RCSA) 22a-449(c) 100 through 110] regulations.

QC sampling must meet normal laboratory precision and accuracy limits. QC samples include matrix spike/matrix spike duplicates and temperature blanks collected at the frequencies presented in Worksheet #20. Non-detect results will be rejected if the total solids content in the associated sediment sample is less than 30 percent. Because these data will be used for waste characterization purposes and not human health risk assessment, limited validation will be performed using method-specific criteria, and the logic for data qualification detailed in Region 1 EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses will be used to apply qualifiers to data.

11.3 STUDY BOUNDARIES

The in-situ characteristics of the contaminated sediment in Inner Pier 1 must be determined to identify appropriate disposal options. The physical boundaries that define the lateral extent of Inner Pier 1 are illustrated on Figure 10-2. Shallow bedrock provides the vertical physical constraint that restricts sediment sampling below approximately 6 feet in Inner Pier 1.

Surface sediment can have the highest contaminant concentrations if contamination was recently deposited at a site or if a depositional environmental does not exist. Surface sediments from 0 to 6 inches are typically defined as the biologically active zone. For this project, surface sediment is defined as the top 1 foot of sediment. Sediment samples will be collected from this interval, and the resulting sediment concentrations will be compared to the following criteria:

- TSCA (40 CFR 761) threshold limit of 50 mg/kg, above which the sediment will need to be managed/disposed of as TSCA waste.

- Characteristic hazardous waste limits for metals per 40 CFR 261 and RCSA 22a-449(c)-101 (see Worksheet 15), above which the sediment will need to be managed, treated, and/or disposed of as hazardous waste.

Deeper sediment can have high contaminant concentrations if the contamination was historically deposited and a depositional environment exists. Characterizing the 2 to 4 feet below sediment surface (bss) sediments will be sufficient for this project because shallow bedrock limits the vertical extent of sediment. Sediment data from the 2- to 4-foot interval will be compared to the same criteria used to evaluate surface sediment.

In summary, sediment depth intervals of 0 to 1 foot and 2 to 4 feet bss are considered sufficient for purposes of in-situ characterization. The results of the proposed sediment sampling program will be used to define the in-situ characteristics of the contaminated sediment for disposal purposes.

11.4 ANALYTICAL APPROACH

The decision rule below defines how the data will be used.

Decision Rule #1: See Figure 11-1. For the study area, if measured PCB Aroclor and TCLP metals concentrations in all collected sediment samples meet the disposal requirements for placement in a solid waste landfill (see Section 11.3 for criteria), then recommend disposal in a solid waste landfill. Otherwise, recommend evaluation of other disposal alternatives based on the observed contaminant concentrations, contaminated waste volumes, and waste acceptance criteria of available disposal facilities. The project team will strive to minimize waste volumes while ensuring that disposal is cost-effective and timely.

Additional waste characterization activities will be required after the sediment is dredged but before it is disposed. The contractor performing the sediment dredging and waste disposal activities will work with the project team to decide how this will be handled based on characterization results and likelihood of different disposal options. This information will be included in the remedial action work plan. Various factors based on lines of evidence will be used to ensure that project objectives are attained.

11.5 PERFORMANCE OR ACCEPTANCE CRITERIA

The project team allocated a relatively small number of samples as efficiently as possible with the resources available. The samples were allocated using a judgmental sampling design based on knowledge of past spatial coverage by samples and knowledge of site operations. The intent is to efficiently characterize in-situ contaminant concentrations in the sediment of Inner Pier 1 while achieving spatial coverage sufficient to fill data gaps. Because the sampling design is judgmental, no statistical computation of the required number of samples and no specification of statistically based decision performance objectives were necessary. Direct comparison of measured concentrations to the project criteria (waste identification criteria) will be straightforward.

11.6 PLAN DEVELOPMENT FOR OBTAINING DATA

To complete the in-situ characterization of the Inner Pier 1 sediment, sediment cores will be collected from seven locations. Sediment samples will be collected from the cores at one or two depth intervals (0 to 1 foot bss and from 2 to 4 feet bss) depending on the sample location. Three cores (TRP1-SD-011 through TRP1-SD-013) will be collected at the northern end of Inner Pier 1 from locations not previously sampled. Historical sampling has shown that sediment in this area has high contaminant concentrations (PAHs, PCBs, and metals). Sediment cores to 4 feet bss will be collected at these locations to allow for collection of sediment samples from both sample intervals. If sediment cores cannot be collected to 4 feet bss at these locations because of shallow bedrock, a deep sediment sample (2 to 4 feet bss) will be collected at one of the four other proposed sediment sample locations (TRP1 SD-014 through TRP1-SD-017), as needed. The four additional cores (TRP1 SD 014 through TRP1-SD-017) will be collected at the northern and central portions of Inner Pier 1 near historic sampling locations that had elevated contaminant concentrations. Even though sediment samples are only required to 1 foot bss at these four locations, core samples to 4 feet bss will be collected at these four locations in the event that deeper samples are required from these locations as discussed above. The sampling program will result in a total of seven surface (0 to 1 foot bss) and three subsurface samples (2 to 4 feet bss). Worksheet #17 provides a detailed discussion on the design and rationale of the field sampling plan. The samples will be analyzed as appropriate for PCB Aroclors and TCLP metals. Figure 10-2 shows the proposed Inner Pier 1 sample locations.

SAP Worksheet #12 -- Measurement Performance Criteria Table
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Cooler Temperature Blank	All Fractions	One per cooler	Accuracy / Representativeness	Between 2 and 6 ° C	S&A

Worksheet #20 provides rationale for not collecting field quality control samples.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation/ collection dates)	How Data Will Be Used	Limitations on Data Use
Marine Railway Investigation	SAIC Evaluation of Chemical Data for the Pier 1 Marine Railway 2000	SAIC Sediment samples 1999	Data will be used with new data collected for this project to characterize in-situ sediment for waste disposal purposes.	Surface grab sediment samples were collected in Inner Pier 1, but no core samples were collected during the investigation. Although more than 10 years old and potentially not completely representative of current conditions, the data will be assumed to be representative unless gross anomalies (e.g., in spatial concentration patterns) are detected.
Pilot Study	Battelle Final Thames River Rapid Sediment Characterization Pilot Study Survey Report 2003	Battelle Sediment samples 2003	Data will be used with new data collected for this project to characterize in-situ sediment for waste disposal purposes.	Surface grab sediment samples were collected adjacent in Inner Pier 1, but no core samples were collected during the investigation. Data are only useful for estimating characteristics in surface sediments.

Secondary Data	**Data Source** (originating organization, report title and date)	**Data Generator(s)** (originating organization, data types, data generation/ collection dates)	**How Data Will Be Used**	**Limitations on Data Use**
Thames River Validation Study	Battelle/Neptune & Company Thames River Validation Study Report March 2008	Battelle/Neptune & Company. Chemical data for sediment (2004)	Data will be used with new data collected for this project to characterize in-situ sediment for waste disposal purposes.	The Validation Study included grab sediment samples that were generally limited to the top 5 cm of sediment and core samples that were generally limited to 6 feet bss. Data are useful for estimating characteristics in surface and subsurface sediments.

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

The project tasks include the following:

- Mobilization/Demobilization
- Vibracore Sediment Sampling
- QC Tasks
- Equipment Decontamination
- Investigation-Derived Waste Management
- Analytical Tasks
- Data Management
- Assessment and Oversight
- Data Review
- Project Report

The Standard Operating Procedures (SOPs) and field documents referenced below and in the various worksheets are included in Appendices A and B, respectively, on the attached CD.

- **Mobilization/Demobilization** - A field team orientation meeting will be conducted prior to the start of fieldwork to familiarize the team personnel with the site's health and safety requirements, objectives and scope of the field activities, and chain-of-command. This meeting will be attended by the FOL/SSO, PM, and lead chemist. Mobilization activities will include transporting field personnel, equipment, supplies, and the boat subcontractor to the site. One mobilization is planned for August 2009. All coring equipment and sampling tools will be cleaned prior to arrival on site. A 1-hour health and safety meeting will be conducted prior to initiating onsite activities. All subcontractor personnel (including substitutes) will attend the meeting. Tetra Tech will coordinate with the Navy Point of Contact (POC) at NSB-NLON (Environmental Restoration Program Manager) regarding passes, security and access issues, and daily activities. Tetra Tech will also coordinate with the Navy RPM and stakeholders regarding the field activities. Demobilization will include transporting personnel, field equipment, supplies, and the boat subcontractor from the site, performing general site cleanup, and organizing and finalizing field paperwork.

- **Vibracore Sediment Sampling** - The Tetra Tech FOL/SSO and boat subcontractor (captain and one crew member) will perform vibracore sediment sampling. The Navy POC at NSB-NLON will pre-arrange site access with the Port Operations and Security Departments at NSB-NLON. The boat captain will provide daily plans to the Port Operations and Security Departments. The subcontractor will launch their boat at the Navy launch facility at Pier 1 and navigate to the study area. The boat will be navigated by the captain to the sampling locations using pre-determined coordinates and a differential Global Positioning System (GPS). The coordinates for each sample location are provided in the following table.

Sample Location ID	Northing (NAD 83)	Easting (NAD 83)	Latitude	Longitude
TRP1-SD-011	703822.333	1181222.657	41° 23' 26.98''	72° 05' 22.36''
TRP1-SD-012	703792.013	1181199.918	41° 23' 26.68''	72° 05' 22.66''
TRP1-SD-013	703793.273	1181249.197	41° 23' 26.69''	72° 05' 22.01''
TRP1-SD-014	703808.633	1181230.237	41° 23' 26.85''	72° 05' 22.26''
TRP1-SD-015	703816.023	1181254.247	41° 23' 26.92''	72° 05' 21.94''
TRP1-SD-016	703755.372	1181230.238	41° 23' 26.32''	72° 05' 22.26''
TRP1-SD-017	703730.832	1181240.688	41° 23' 26.08''	72° 05' 22.13''

Upon arrival at a sample location, the boat will be anchored to remain over the desired location, and one 4-foot-long sediment core will be collected using the vibracore method. A 3-inch-diameter stainless steel core barrel with a cutting edge and retaining fingers at the lower end will be used to collect the cores. A 2⅝-inch polycarbonate tube will be included inside of the core barrel to contain the sample after collection. Core samples will be collected at seven locations at Inner Pier 1 (Figure 10-2). If the boat and sampling equipment are unable to access the locations or reach the desired depths, the locations will be moved in the field only a distance necessary to provide adequate access or depth but remaining as close to the original locations as possible. After retrieval of the cores to the surface, the core tubes will be removed from the core barrel and secured. Excess water at the top of the core will be drained. The core tubes will be transported onshore and opened at a designated work station. After opening, each sediment core will be photographed and thoroughly described on a boring log by a Tetra Tech geologist. Sampling intervals will depend on the sample location. Sample intervals for three locations (TRP1-SD-011 through TRP1-SD-013) will consist of the following: 0 to 1 foot bss and 2 to 4 feet bss. Sample intervals for the four remaining locations (TRP1-SD-014 through TRP1-SD-017) will be 0 to 1 foot bss. If any or all of the deeper samples cannot be collected at the three designated locations because of encountering bedrock refusal prior to reaching approximately 3.5 feet bss, deeper samples will be collected from one of the other four sample locations. The volume of sediment required per interval sampled is approximately 20 ounces to complete the required analyses. Sample material will be homogenized in disposable bowls with disposable trowels. After homogenization, the disposable trowels will be used to place the sediment in the appropriate sample bottles. The bottles will be secured after filling and then placed on ice in coolers. The coolers will be secured for shipment to the laboratory. Additional information regarding sediment sampling procedures is provided in the Tetra Tech SOPs SA-1.2 and SA-6.1 and TG&B SOP – Vibrocoring Procedure included in Appendix A on the attached CD.

- **QC Tasks** - The only field QC samples for the project will be temperature blanks. These blanks will be used by the laboratory to confirm that the samples were cooled to the proper temperature. Also, additional sample volume will be collected for the laboratory to perform matrix spike/matrix spike duplicate analysis. Worksheet #20 presents the field QC sample summary.

- **Equipment Decontamination** - The decontamination procedures presented in Tetra Tech SOP SA-7.1 in Appendix A on the attached CD will be followed for this project. The core barrel and cutting head will be decontaminated between uses at the sample locations. The core barrel will be scrubbed cleaned of all visible debris, sprayed with a soap and water solution, and rinsed in river water. The dilute decontamination fluids will be discharged to the Thames River. To minimize decontamination, new disposal trowels and bowls will be used to process each sediment sample and will be discarded after one use. Therefore, decontamination of this equipment will not be necessary. Personal protective equipment and emergency decontamination procedures are discussed in the HASP.

- **Investigation-Derived Waste Management** - Disposable core liners, used personal protective equipment, and general project refuse will be collected and placed in plastic bags (doubly lined) and disposed in a Navy-approved trash receptacle. Waste water generated during decontamination of vibracore sampling equipment will be discharged into the Thames River.

- **Analytical Tasks** - Chemical analysis of the sediment samples for PCBs and TCLP metals will be performed by Mitkem, the subcontracted laboratory. Analyses will be performed in accordance with the analytical methods identified in Worksheet #18 and the requirements of the technical specification for laboratory services developed by Tetra Tech for this work. The technical specification is included as Appendix C. The subcontracted laboratory will meet the QLs and Method Detection Limits (MDLs) specified in Worksheet #15.

  The technical specification details the analytical requirements, number of samples, matrix, analytical methods to be performed, preservatives, holding times, QLs required for the project, and data deliverables. Mitkem will perform the chemical analyses following laboratory-specific SOPs developed based on the methods listed in Worksheet #23. Copies of the laboratory SOPs are included in Appendix D on the attached CD.

- **Data Management** - Project data will be managed according to the procedures outlined on the following worksheets:

  - Project documentation and records
    - Field sample collection and field measurement records - See Worksheets #27 and #29
    - Laboratory data package deliverables - See Worksheet #30
    - Data assessment documents and records - See Worksheet #29

  - Data recording formats - See Worksheet #27

  - Data handling, management, tracking, and control

Data Handling and Management - After the field investigation is completed, field sampling log sheets will be organized by date and medium and filed in the project files. The field logbooks for this project will be used only for this site and will also be categorized and maintained in the project files after completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratory (Mitkem) will meet the requirements of the technical specification (see Appendix C). The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

Data Tracking and Control - The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking**. Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contract laboratory. Upon receipt of the data packages from the analytical laboratory, the Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.

- **Data Storage, Archiving, and Retrieval**. The data packages received from the subcontract laboratory are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample log sheets, and chain-of-custody records will be submitted by the FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.

  Data Security. The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

- **Assessment and Oversight** - See Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

- **Data Review -** Data reviews will be conducted in accordance with the procedures outlined on the following worksheets:

- Data verification – See Worksheet #34
- Data validation – See Worksheets #35 and #36
- Usability assessment – See Worksheet #37

- **Project Report** - The analytical data generated under this SAP will be used for the following purposes:

  - To develop an EE/CA, Action Memorandum, and Removal Action Work Plan for Inner and Outer Pier 1.

Draft versions of these documents will be prepared and submitted in hardcopy format to the Navy, EPA, CTDEP, and NOAA for review and comment. Tetra Tech will develop responses to comments received on the draft reports. The final versions of the reports will submitted in hardcopy and electronic format to the project team.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment
Analytical Group: PCB Aroclors

Analyte	CAS Number	Project Action Limit[1] (µg/kg)	Project Action Limit Reference[2]	Project Quantitation Limit Goal (µg/kg)	Mitkem[3,4]	
					QL	MDL
Aroclor-1016	12674-11-2	7143	TSCA	333	33	4.7
Aroclor-1221	11104-28-2	7143	TSCA	333	33	2.8
Aroclor-1232	11141-16-5	7143	TSCA	333	33	4.7
Aroclor-1242	53469-21-9	7143	TSCA	333	33	4.5
Aroclor-1248	12672-29-6	7143	TSCA	333	33	4.9
Aroclor-1254	11097-69-1	7143	TSCA	333	33	2
Aroclor-1260	11096-82-5	7143	TSCA	333	33	6.2

1 The overall Project Action Limit for PCBs is 50 mg/kg, which is for the sum of all PCB Aroclors. The Project Action Limits provided for the individual Aroclors are 1/7 of overall Project Action Limit.
2 TSCA 40 CFR Part 761.
3 Mitkem-specific MDLs and QLs are presented.
4 QL/MDL values are based on 100 percent solid samples.

Matrix: Aqueous leachate
Analytical Group: Metals

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/L)	Mitkem[2]	
					QL	MDL
Silver	7440-22-4	5000	40 CFR 261.11	1670	30	0.59
Chromium	7440-47-3	5000	40 CFR 261.11	1670	20	1.1
Cadmium	7440-43-9	1000	40 CFR 261.11	333	5	0.14
Lead	7439-92-1	5000	40 CFR 261.11	1670	10	2.2
Barium	7440-39-3	100000	40 CFR 261.11	3300	200	8.5
Arsenic	7440-38-2	5000	40 CFR 261.11	1670	20	5.3
Selenium	7782-49-2	1000	40 CFR 261.11	333	30	6.6
Mercury	7439-97-6	200	40 CFR 261.11	66	0.2	0.016

1 40 CFR Part 261.11 and RCSA 220-449(c)-101 (references 40 CFR Part 261.11)
2 Mitkem-specific MDLs and QLs are presented.

SAP Worksheet #15B -- Reference Limits and Evaluation Table – Backup criteria

(UFP-QAPP Manual Section 2.8.1)

No additional values were considered in evaluating detection limits.

SAP Worksheet #16 -- Project Schedule/Timeline Table (optional format)
(UFP-QAPP Manual Section 2.8.2)

Activity	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Internal Draft UFP-SAP	Tetra Tech	5/13/09	6/17/09	Internal Draft UFP-SAP	6/17/09
Navy Review	Navy	6/18/09	6/24/09	NA	NA
Resolve Comments	Tetra Tech	6/25/09	6/29/09	NA	NA
Draft UFP-SAP	Tetra Tech	6/30/09	7/1/09	Draft UFP-SAP	7/1/09
Regulator Review	EPA and CTDEP	7/2/09	8/13/09	NA	8/13/09
Comment Resolution	Tetra Tech	8/14/09	8/20/09	Responses to Comments	8/20/09
Final UFP-SAP	Tetra Tech	8/21/09	9/7/09	Final UFP-SAP	9/7/09
Field Work and Analysis	Tetra Tech	9/8/09	10/9/09	NA	NA
Data Validation	Tetra Tech	10/10/09	10/26/09	Final Analytical Data Tables	NA

SAP Worksheet #17 -- Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

The sampling design for this project is judgmental. A small number of sample locations was selected to fill data gaps for waste disposal determination. The selected number of samples was the maximum that could be selected with available resources, even with substitution of field QC samples by additional site characterization samples. Sediment was sampled during the Marine Railway Investigation, Pilot Study, and Thames River Validation Study. Based on these previous sampling events, sediment samples will be located within the northern end of Inner Pier 1 in areas that had previously high contaminant concentrations in sediment.

Medium and Analyses Rationale

This SAP covers the collection and analysis of sediment from the Thames River adjacent to NSB-NLON in Inner Pier 1. Data collected during the Marine Railway Investigation, Pilot Study, and Thames River Validation Study Thames River Validation Study determined that sediment contained elevated levels of PCBs and metals. Although surface water is a transport mechanism for suspended particulates, it was not found to be a medium of concern in the study area.

To evaluate disposal options, sediment will be tested by the TCLP for specific metals to determine if dredged sediment may be classified as a characteristic hazardous waste. Toxicity characterization can include analysis for select metals, pesticides, herbicides, volatile organic compounds (VOCs), and semi-volatile organic compounds (SVOCs). However, based on a review of existing data, VOCs, SVOCs, pesticides, and herbicides are not present in study area sediment at concentrations that would likely result in the sediment being classified as characteristic hazardous waste. Therefore, the sediment samples will not be analyzed for TCLP VOCs, SVOCs, pesticides, or herbicides. Comparison of maximum metals concentrations detected in Validation Study sediment samples to regulatory leachate levels by the 20 times rule (total mass concentration of metal divided by 20 and then compared to the regulatory leachate concentration) indicates that metals are possibly present in sediment at concentrations that may result in the classification of dredged sediment as a characteristic hazardous waste. Therefore, the sediment samples will be analyzed for TCLP metals. The existing data indicate that the sediment contains PCBs; therefore, the sediment will also be tested for PCBs (Aroclors) to determine if dredged sediment is a TSCA waste (i.e., PCB concentration greater than 50 mg/kg).

Sample Location Rationale

Inner Pier 1 area sediment has been determined to be contaminated and will be removed by a NTCRA. Historical samples collected in the Inner Pier 1 area during the Marine Railway Investigation, Pilot Study, and Validation Study had high concentrations of PCBs and metals. Three additional samples will be collected at the northern end of Inner Pier 1 to verify that PCB concentrations in these previously unsampled locations are less than the TSCA threshold value. In addition to PCBs, the subsurface

samples from these locations will also be analyzed for TCLP metals. A representative number of TCLP metals samples are needed to characterize the subsurface sediment. Historical surface sediment samples from locations P1-48, P1, P1-C1, and SBP-1 had PCB concentrations ranging from approximately 5 to 19 mg/kg. Additional surface sediment samples will be collected from 0 to 1 foot bss in close proximity to these locations and analyzed for TCLP metals to characterize the surface sediment.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (feet bss)	Analytical Group	Number of Samples	Sampling SOP Reference[1]
TRP1-SD-011/TRP1-SD-011-0001	Sediment	0-1	PCBs (Aroclors)	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-011/TRP1-SD-011-0204	Sediment	2-4[2]	PCBs (Aroclors) and TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-012/TRP1-SD-012-0001	Sediment	0-1	PCBs (Aroclors)	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-012/TRP1-SD-012-0204	Sediment	2-4[2]	PCBs (Aroclors) and TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-013/TRP1-SD-013-0001	Sediment	0-1	PCBs (Aroclors)	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-013/TRP1-SD-013-0204	Sediment	2-4[2]	PCBs (Aroclors) and TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-014/TRP1-SD-014-0001	Sediment	0-1	TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-015/TRP1-SD-015-0001	Sediment	0-1	TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1

Sampling Location / ID Number	Matrix	Depth (feet bss)	Analytical Group	Number of Samples	Sampling SOP Reference[1]
TRP1-SD-016/TRP1-SD-016-0001	Sediment	0-1	TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1
TRP1-SD-017/TRP1-SD-017-0001	Sediment	0-1	TCLP metals	1	CT-04, SA-1.2, SA-6.1, SA-6.3, SA-7.1

1 SOP or worksheet that describes the sample collection procedures. SOPs are provided in Appendix A.

2 If any or all deep sediment samples cannot be collected at these locations because of encountering bedrock refusal prior to reaching approximately 3.5 ft bss, collect a deep sediment sample at TRP1-SD-014, TRP1-SD-015, TRP1-SD-016, or TRP1-SD-017.

SAP Worksheet #19 -- Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference[1]	Container (number, size, and type)	Sample volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[2] (preparation / analysis)
Sediment	Total PCB (Aroclors)	SW-846 8082: Lab SOPs 3, 5, 6	8 oz amber glass jar	60 grams	4 ± 2 degrees C	14 days to extraction/ 40 days to analysis
Sediment leachate	TCLP Metals	SW-846 1311/6010/7470 series: Lab SOPs 1, 2, 4, 7	8-oz amber glass jar/Leachate prepared at laboratory	100 grams	4 ± 2 degrees C	28 days to leach (Hg), 6 months (ICP) to leach/28 days to analysis (Hg), 6 months (ICP) to analysis

1 See the Analytical SOP References Table (Worksheet #23).
2 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
NA Not applicable.

Note - Sediment for PCBs and TCLP metals analyses can all be collected into a single 8-oz jar, which typically contains approximately 300 grams of soil. If the sediment appears to contain more than 50 percent water, two 8-oz jars should be collected.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

No trip blanks will be required for this investigation because no samples are being collected for VOC analysis.

No rinsate blanks will be required because disposable equipment will be used for sampling.

Field duplicate samples also will not be collected because it was concluded that collection of field duplicates would not add measurably to project quality. No field duplicates will be required because the heterogeneity of the sediments is more likely to contribute to the variability between the duplicate results rather than the actual collection of the sample. The field duplicates were replaced with additional site characterization samples. In this case, the limited number of QC samples that could be collected will add little understanding to the quality of the data. An additional characterization sample, however, will add significantly to the understanding of contaminant distribution and the concentration levels of contaminants at the site.

All samples designated for chemical analysis will be put in coolers with ice to cool the samples to 4º C. Temperature blanks will be added to each cooler to verify that samples were stored at the correct temperature during shipment. These blanks will be used to help qualitatively determine the representativeness and comparability of the data as well as any biases or imprecision that might result from deviations in storage requirements. Elevated storage temperatures can cause loss of target analytes (e.g., via microbial degradation). Please note that precision and accuracy will be determined using laboratory duplicates, laboratory control spikes/laboratory control spike duplicates (LCS/LCSD), and matrix spike/matrix spike duplicates (MS/MSDs) as per Worksheet #28.

Matrix	Analytical Group	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSD[2] Samples	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples[3]	Total No. of Samples to Lab
Sediment	PCBs (aroclors)	6[4]	0	2	0	0	0	0	8
Sediment	TCLP metals	7[4]	0	0	0	0	0	0	7

1 If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.
2 Although the MS/MSD is not typically considered a field QC sample, it is included here because location determination is often established in the field.
3 The number of batch or project-specific proficiency testing (PT) samples are optional but highly recommended.
4 Not all analyses are performed on every sample. Refer to Worksheet #18 for analyses per sample location.

SAP Worksheet #21 -- Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature	Tetra Tech	NA	Y	
SA-6.3	Field Documentation	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	Y	
SA-6.1	Non-Radiological Sample Handling	Tetra Tech	Sample Bottleware, Packaging Material, Shipping Materials	Y	
SA-1.2	Surface Water and Sediment Sampling	Tetra Tech	Sampling Procedures, Methods	Y	
SA-7.1	Decontamination of Field Equipment	Tetra Tech	Decontaminate core barrel and cutting head (scrub brushes, phosphate-free detergent, river water rinse)	Y	
NA	Vibracoring Procedure	TG&B (Tetra Tech Subcontractor)	Vibracore sampling equipment (BH-5 pneumatically driven system, air compressor, core barrel, cutting head)	Y	

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

No field equipment requiring calibration will be used.

SAP Worksheet #23 -- Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
1	100.0111: Metals by Method 6010C, Rev 11, 11/08	Definitive	Leachate, ICP Metals	ICP	Mitkem	N
2	100.0012: Mercury by Method 7470/747, Rev 9, 1/08	Definitive	Leachate, Mercury	CVAA	Mitkem	N
3	60.0003: PCBs by Method 8082, Rev. 8, 9/08	Definitive	Sediment, PCB Aroclors	GC/ECD	Mitkem	N
4	110.0025:TCLP by Method 1311, Rev 5, 7/04	Definitive	Sediment, TCLP	NA	Mitkem	N
5	50.0052, Organic Preparation of Soil Samples by Sonication, Method 3550, Rev. 2, 1/08	Definitive	Sediment, PCBs	NA	Mitkem	N
6	50.0100, Organic Preparation of Soil Samples by MSE, Method 3570, Rev. 1, 1/08	Definitive	Sediment, PCBs	NA	Mitkem	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
7	100.0003, Sample Preparation of Aqueous Samples by Acid Digestion for ICP, Method 3005/3010, Rev. 7, 1/09	Definitive	Leachate, ICP Metals	NA	Mitkem	N
8	20.0003, Logging work orders into Omega LIMS Rev. 3, 1/08	Definitive	All Laboratory Analyses	NA	Mitkem	N
9	30.0003, Sample Receipt, Storage, Tracking and Disposal, Rev. 12, 1/08	Definitive	All Laboratory Analyses	NA	Mitkem	N
10	30.0024, Sample Disposal, Rev. 8, 1/08	Definitive	All Laboratory Analyses	NA	Mitkem	N
11	110.0026, Handling of Evidentiary Materials, Rev. 6, 1/06	Definitive	All Laboratory Analyses	NA	Mitkem	N
12	110.0035, Sample Data Control, Rev. 1, 1/08	Definitive	All Laboratory Analyses	NA	Mitkem	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
13	110.0034, Sample Data Control for Inorganic CLP, Rev. 1, 3/09	Definitive	All Laboratory Analyses	NA	Mitkem	N

1 If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project-specific needs must be reviewed and approved by the Navy QAO.

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/ECD	Initial Calibration Continuing Calibration	After major instrument maintenance. Every 12 hours and at end of sequence.	%Relative Standard Deviation <20% %Difference <20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	Lab SOP 4
CVAA	Initial Calibration Continuing Calibration	Every analytical run. Every 10 samples and at end of run.	Initial Calibration: correlation coefficient >=0.995 Continuing Calibration: within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	Lab SOP 2
ICP	Initial Calibration	The instrument is calibrated at the beginning of each day or if the QC is out of criteria.	The instrument is calibrated by a three-point calibration. The minimum correlation coefficient for the curve is 0.998.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	Lab SOP 1
	Initial Calibration Blank (ICB)	After every initial calibration.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze.	Analyst/Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	Continuing Calibration (CCV)	Analyze a standard after every 10 samples and at the end of the sequence.	The acceptance criterion for the continuing calibration standard is 90-110% recovery of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	
	Continuing Calibration Blank (CCB)	After every continuing calibration verification.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst/Supervisor	

1 See Analytical SOP References table (Worksheet #23).

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/ECD	Check for leaks, replace gas line filters, recondition or replace column, clean injection port/liner.	Monitor instrument performance via CCV.	Monitor instrument performance via CCV.	Daily, after every 10 samples.	%Difference < = 20%	Replace connections, replace gas line filters, replace GC column, clip column, replace injection port liner, clean injection port.	Mitkem Department Supervisor	Lab SOP 3
ICP	Perform leak test, change pump tubing, change torch and window, clean filters.	Monitor instrument performance via CCV and CCB.	Monitor instrument performance via CCV and CCB.	Daily, after every 10 samples.	All analytes within +/- 10%, no analytes > quantitation limit.	Replace pump tubing, replace torch and window, clean all filters.	Mitkem Department Supervisor	Lab SOP1
CVAA	Perform leak test, change tubing, clean window, clean filters.	Monitor instrument performance via CCV and CCB.	Monitor instrument performance via CCV and CCB.	Daily, after every 10 samples.	CC: within +/- 10%, Hg not > quantitation limit.	Replace connections, replace pump tubing, clean all filters.	Mitkem Department Supervisor	Lab SOP 2

1 Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

SAP Worksheet #26 -- Sample Handling System
(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL (TBD)/Tetra Tech
Sample Packaging (Personnel/Organization): FOL (TBD)/Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL (TBD)/Tetra Tech
Type of Shipment/Carrier: Laboratory courier service (Mitkem)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff (organic/inorganic), Mitkem Laboratories
Sample Determinative Analysis (Personnel/Organization): GC/MS, GC, Metals Laboratory Staff, Mitkem Laboratories
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 1
Sample Extract/Digestate Storage (No. of days from extraction/digestion) : 6 months from submittal of final data report
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian, Mitkem Laboratories
Number of Days from Analysis: 60 days from submittal of final data report

SAP Worksheet #27 – Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

SAMPLE DESIGNATION AND TRACKING SYSTEM

Each sample collected will be assigned a unique sample tracking number used to catalog the associated results. The sample tracking number will consist of alpha-numeric characters identifying the site, sample medium, location, core, and depth. Any other pertinent information regarding sample identification will be recorded on the sample log sheets or in the field logbook. The alpha-numeric (A-N) coding to be used in the sample system is described below.

AAAA	-	AA	-	NNN	-	A	-	(NNNN)
(Site ID)	-	(Medium)	-	(Location)	-	(Core)	-	(Depth)

Site identifier: TRP1 for Inner Pier 1

Medium identifier: "SD" for sediment samples

Sample location identifier: Each sediment sample location will be assigned a unique location identifier.

Sample core identifier: One core will be taken at each location. Samples from 0 to 1 foot and 2 to 4 feet will be taken as appropriate from the core sample.

Depth: For sediment samples, this portion of the sample tracking number will represent the depth of the sample bss, "0001".

Example: A sediment sample collected from Inner Pier 1, location 011, and sample interval 0 to 1 foot bss, will be identified as: TRP1-SD-0011-0001.

QC samples collected during a sampling program typically use the same coding system as the environmental samples; however, no field QC samples will be collected for this project. Laboratory QC samples (MS/MSD samples) have no separate sample identifier codes but will be designated on the chain-of-custody record and sample log sheet.

SAMPLE COLLECTION DOCUMENTATION

A project-specific field logbook will be used to keep daily records of significant events, observations, and measurements during field investigations. The field logbook also will be used to document all sampling

activities. Logbook entries will be made with indelible ink to provide a permanent record, and any errors in the logbook will be verified, crossed through, and initialed by the person discovering the error. Field logbooks will be maintained according to Tetra Tech SOP No. SA-6.3 (Appendix A). The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during field activities. Field logbooks should be permanently bound and pre-paginated; designated forms should be used whenever possible to ensure that field records are complete. The following items are examples of information that may be included in a field logbook:

- Name, date, and time of entry
- Names and responsibilities of field crew members
- Name and titles of any site visitors
- Descriptions of field procedures and problems encountered
- Samples collected at each location
- Details of sampling location, including sampling coordinates
- Sample identification numbers of all samples collected
- Date and time of collection
- Sample collector
- Sample collection method
- Decontamination procedures
- Weather conditions
- Site observations
- Site sketches
- Health and safety issues including personal protective equipment
- Log of photographs

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. The following sections outline the information that will be documented in the field according to the medium to be sampled and the activities to be performed. Examples of these forms can be found in Appendix B.

FIELD SAMPLE HANDLING AND CHAIN-OF CUSTODY PROCEDURES

Custody of samples must be maintained and documented at all times to ensure the integrity of each sample from collection through analysis. An accurate written record is necessary to trace the possession and handling of the sample; this documentation is referred to as the "chain-of-custody" form. Chain of custody begins when samples are collected in the field and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be delivered to the laboratory accompanied by a chain-of-custody form that will describe the sample identifiers, dates and times of sample collection, analytical parameters, and persons responsible for the sample integrity.

Prior to sample collection, sample containers will be labeled with the sample location number, sampler's name, date, and analytical fraction. Following collection, samples will be placed on ice in a secure cooler

and attended by Tetra Tech personnel or placed in locked vehicles or designated storage areas until shipment to an off-site laboratory. Chain-of-custody procedures are described in further detail in Tetra Tech SOPs SA-6.3, Field Documentation, and SA-6.1, Non-Radiological Sample Handling (Appendix A).

The samples will be shipped to the laboratory in coolers packed with bubble wrap, or equivalent packing material, to cushion the samples and prevent breakage. Ice will be added to the coolers to maintain the required temperature (4° ± 2°C) of the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with signed custody seals to ensure that chain of custody is maintained. Samples will be shipped to the laboratory by their courier service to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times for each analysis are presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

Each sample collected will be assigned a unique sampling tracking number, as described above. The sample number, sample collection date and time, and a list of the sample analyses to be performed will be recorded on each container and also on the chain-of-custody form. The chain-of-custody form is a two-part form, the original accompanies the samples to the analytical laboratory, and the copy will be archived in the project files. The following information will be recorded on the chain-of-custody form:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- QC sample designation
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

The field crew will attempt to identify any potentially high concentration samples on the chain-of-custody form.

LABORATORY CUSTODY PROCEDURES

Mitkem SOPs 30.0003 Rev 12, 1/08, Sample Receipt, Storage, Tracking and Disposal; 110.0026 Rev 6, 1/06; Handling of Evidentiary Materials; 20.0003 Rev 3, 1/08, Logging work orders into OMEGA LIMS;

and 110.0027 Rev 8, 4/08, Documentation Policy and Procedures, will be followed. The following text summarizes these SOPs.

The condition of the shipping cooler, custody seals, coolant, integrity and condition of the samples, and presence and accuracy of the chain-of-custody documentation will be recorded upon sample receipt. The Laboratory Sample Custodian will then log the samples into Mitkem's computerized Laboratory Information Management System (LIMS). Any discrepancies are immediately brought to the attention of the Mitkem Project Manager for discussion and resolution with the client. Login information will be distributed electronically to each laboratory section, where supervisors track analyses, holding times, and due dates. Special technical instructions including customized analyte lists, special reporting limits, and any other pertinent information are also contained within the LIMS and are accessible to all staff.

The process of logging samples into LIMS assigns each sample a unique laboratory identification number in ascending sequence. After assignment of sample numbers and required tests for all samples in a submittal, the LIMS generates login paperwork summarizing each project. The Mitkem Project Manager will review the sample receipt and login paperwork to ensure that the login process was performed correctly.

The LIMS sheet tracks the status of each test and work order according to the project due date and lists the number of incomplete samples in each laboratory section's backlog. As work is completed and reviewed for each test, the updated information will be recorded in the LIMS. This ensures visibility for project status and due dates and provides a means of tracking samples through the entire analytical process. This also allows project managers to determine the laboratory's capacity by viewing the backlog of samples in house.

All transfers of samples and sample extracts/digestates will be recorded in custody logs. The entire laboratory facility is maintained as a secure limited-access facility. All documents received with a sample delivery group (SDG) and/or generated in the course of the analyses of samples will be kept confidential. Documentation procedures for record content, format, corrections, dates, and signatures will be implemented to meet the requirements for legally defensible data.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Leachate (TCLP)
Analytical Group	Metals
Analytical Method / SOP Reference	SW-846 6010C and SW7470A/7471A Lab SOPs 1,2

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples.	No target analytes > 1/2 the Quantitation Limit.	Investigate contamination. Redigest and reanalyze, if sample concentration is >10X blank concentration narrate.	Laboratory Supervisor	Bias / Contamination	No target analytes > 1/2 the Quantitation Limit.
Laboratory Control Sample	1 per batch or 1 per 20 samples.	%Recovery 80% - 120%	Redigest and reanalyze. Narrate if recovery is high and sample is <QL.	Laboratory Supervisor	Accuracy / Bias / Contamination	%Recovery 80% - 120%
Duplicate Sample	1 per 20 samples.	RPD <=20%	Qualify Data	Laboratory Supervisor / Data Validator	Precision	RPD <=20%
Matrix Spike	1 per 20 samples.	%Recovery 80% - 120%	Perform post-digestion spike analysis, qualify data.	Laboratory Supervisor / Data Validator	Accuracy / Bias	%Recovery 80% - 120%

Matrix	Leachate (TCLP)					
Post-digestion Spike	For compounds outside of QC limits in Matrix Spike	%Recovery 75% - 125%	Qualify data.	Laboratory Supervisor / Data Validator	Accuracy / Bias	%Recovery 75% - 125%
ICP Serial Dilution	Per analytical run.	%Difference 90% - 110%	Qualify data	Laboratory Supervisor / Data Validator	Accuracy / Bias	%Difference 90% - 110%

Matrix	Sediment
Analytical Group	PCB
Analytical Method / SOP Reference	SW-846 8082/Lab SOP 3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch or 1 per 20 samples.	No target analytes > 1/2 the Quantitation Limit.	Investigate source of contamination. Rerun method blank. Re-extract associated batch of samples unless ND for the contaminant.	Laboratory Supervisor	Bias / Contamination	No target analytes > 1/2 the Quantitation Limit.
Surrogates	Each sample.	Within lab statistical QC limits as reported in the table below.	Unless obvious chromatographic interference, if sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag with * on Form 2.	Laboratory Supervisor / Data Validator	Accuracy / Bias	Within lab statistical QC limits.

Matrix	Sediment					
Laboratory Control Sample	1 per batch or 1 per 20 samples.	Within lab statistical QC limits as reported in the table below.	Reanalyze once. If the LCS recoveries are high but the sample results are <QL narrate. Flag with * on Form 3	Laboratory Supervisor / Data Validator	Accuracy / Bias	Within lab statistical QC limits.
Matrix Spike/ Matrix Spike Duplicate	Every 20 samples.	Within lab statistical QC limits as reported in the table below.	Check instrument performance, qualify data	Laboratory Supervisor / Data Validator	Precision / Accuracy / Bias	Within lab statistical QC limits.

Lab Statistical QC Limits

AT	Analyte	Low	High	RPD
A	Aroclor-1016	40	140	30
A	Aroclor-1260	60	130	30
S	Decachlorobiphenyl	60	125	0
S	Tetrachloro-m-xylene	27	120	0

A = Target Analyte (spiked in LCS/LCSD and MS/MSD)

S = Surrogate

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records • Field logbook (and sampling notes) • Field sample forms (e.g., sample log sheets, drilling logs, etc.) • Chain-of-custody records • Sample shipment airbills • Photographs • Field Task Modification Forms • Sampling and Analysis Plan • Field Sampling SOPs • Boring Logs	Tetra Tech project file, results will be discussed in subject document.
Laboratory Documents and Records • Sample receipt/login form • Sample storage records • Sample preparation logs • Standard traceability logs • Equipment calibration logs • Sample analysis run logs • Equipment maintenance, testing, and inspection logs • Corrective action forms • Reported field sample results • Reported results for standards, quality control checks, and quality control samples • Data completeness checklists • Sample storage and disposal records • Telephone logs • Extraction/clean-up records • Raw data	Tetra Tech Project File, long-term data package storage at third-party professional document storage firm (Business Records Management, inc.), results will be discussed in subject document.
Data Assessment Documents and Records • Field Sampling Audit Checklist (if an audit is conducted) • Analytical Audit Checklist (if an audit is conducted) • Data Validation Memoranda	Tetra Tech project file, results will be discussed in subject document.

SAP Worksheet #30 -- Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Sediment	PCBs (Aroclors)	See Worksheet #18	SW-846 8082:Lab SOP 3	21 days	Ed Lawler Mitkem Corporation 175 Metro Center Boulevard Warwick, RI 02886-1755 (401) 732-3400 ph (401) 732-3499 fax	Not Applicable
Leachate	TCLP Metals	See Worksheet #18	SW-846 1311/6010/7470 series: Lab SOP 1, 2, 4	21 days	Mitkem as above	Not Applicable

SAP Worksheet #31 -- Planned Project Assessments Table
(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Supervision	Daily during sampling events	Internal	Tetra Tech	Tetra Tech FOL	Tetra Tech FOL	Tetra Tech FOL and field crew	Tetra Tech FOL, PM, and Program QAM
Project Supervision	Every sampling event	Internal	Tetra Tech	Tetra Tech PM	Tetra Tech FOL	Tetra Tech PM and FOL	Tetra Tech PM and FOL
Field Sampling System Audit	1 per contract year	Internal	Tetra Tech	Person assigned by Tetra Tech QAM	Tetra Tech PM and FOL	Tetra Tech Auditor and PM	Tetra Tech CLEAN QAM
Laboratory System Audit	Every 18 months	External	Naval Facilities Engineering Service Center (NFESC)	NFESC	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager	NFESC

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Time Frame for Response
Field Supervision	Site logbook and sample collection logs	Corey Rich, PM, Tetra Tech; TBD, FOL, Tetra Tech	Immediately	Entry in site logbook	Corey Rich, PM, Tetra Tech; TBD, FOL, Tetra Tech	24 hours
Project Supervision	Written report	John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Monthly	Written memo	John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Within a week of notification
Field Sampling System Audit (1)	Audit checklist (as per Navy Installation Restoration Chemical Data Quality Manuel [IRCDQM]) and written audit report	Corey Rich, PM, Tetra Tech; TBD, FOL, Tetra Tech; John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Dependent on the finding; if major a stop work may be issued immediately; however, if minor within 1 week of audit	Written memo	Corey Rich, PM, Tetra Tech; TBD, FOL, Tetra Tech; John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Ed Lawler, Mitkem Laboratory Director; Sharon Lawler Mitkem QAM	Not specified by NFESC	Letter	NFESC	Specified by NFESC

1 Audits are scheduled at the Tetra Tech program level and may or may not include this project.

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group (SDG)	Once validation is complete	Tetra Tech DVM or designee	Tetra Tech PM, project file
Major Analysis Problem Identification (internal memo)	When persistent analysis problems are detected	Immediately	Tetra Tech Program QAM	Tetra Tech PM, Program QAM, Program Manager, and project file
Project Monthly Progress Report	Monthly for duration of project	Monthly	Tetra Tech PM	Navy, project file
Field Progress Report	Daily, oral, during the course of sampling	Every day that field sampling is occurring	Tetra Tech FOL	Tetra Tech PM
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately	Laboratory PM	Tetra Tech PM, project file

SAP Worksheet #34 -- Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody forms to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, and the Tetra Tech PM. See SOP SA-6.3.	Internal	Tetra Tech sampler and FOL
SAP Sample Tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	Tetra Tech FOL or designee
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	Tetra Tech FOL or designee
Sample Coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	Tetra Tech FOL or designee
Field QC Samples	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	Tetra Tech FOL or designee
Chain-of-Custody Forms	The laboratory sample custodian will review the sample shipment for completeness, and integrity, and will sign accepting the shipment. The Tetra Tech data validator will check that the chain-of-custody form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses.	External/ Internal	Laboratory sample custodian/ Tetra Tech data validator
Analytical Data Package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package.	External	Mitkem Laboratory QAM
Analytical Data Package	The data package will be verified for completeness by Tetra Tech data validator. Missing information will be requested from the laboratory.	Internal	Tetra Tech data validator

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Electronic Data Deliverables (EDDs)	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the method detection limit and reporting limit will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	Internal	Tetra Tech data validator

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs/Field Logs/Sample Collection Logs	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken chain of custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Tetra Tech PM, FOL, or designee
IIa	Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Laboratory QAM (Mitkem)
IIa	Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the laboratory will contact Tetra Tech for guidance prior to report preparation.	Laboratory QAM (Mitkem)
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded.	Tetra Tech Project Chemist or data validator
IIa	Holding Times	Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Tetra Tech Project Chemist or data validator
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPC, listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tetra Tech Project Chemist or data validator

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa/IIb	Laboratory Duplicate Analyses for Precision	Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses, MS/MSDs, and LCS/LCSDs. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet #12.	Tetra Tech Project Chemist or data validator
IIa/IIb	Sample Results for Representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	Tetra Tech Project Chemist or data validator
IIa/IIb	PALs	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminants, on the other target compounds reported as no-detected. Document this usability issue and inform the PM.	Tetra Tech Project Chemist or data validator
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifier codes that summarize the reasons for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Tetra Tech Project Chemist or data validator
IIa, IIb	SAP QC Sample Documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall have contacted the Tetra Tech PM.	Tetra Tech PM or designee

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa, IIb	Documentation of Analytical Reports for Completeness	Review the chain-of-custody form generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Tetra Tech Project Chemist or data validator
IIa/IIb	PALs	Review and add PALs to the laboratory EDD. Flag samples and notify PM of samples that exceed PALs, as listed on Worksheet #15.	Tetra Tech PM or designee
IIb	QLs for Sensitivity	Ensure that the QLs listed in Worksheet #15 were achieved.	Tetra Tech Project Chemist or data validator
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results.	Tetra Tech Project Chemist or data validator

1 IIa=compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP manual, V.1, March 2005].
IIb=comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP manual, V.1, March 2005].

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table
(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / IIb	Sediment	PCBs (Aroclors)	EPA Region 1 Tier III data validation. Criteria for SW-846 8082 listed in Worksheets #12, #15, #14, and #28. If not included in Worksheet #12, #25, #24, or #28, to the logic outlined in Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Organic Analyses, Part III, February 2004, will be used to apply qualifiers to data.	Tetra Tech Data Validation Coordinator (Joseph Samchuck) and staff chemists

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / IIb	Aqueous	TCLP Metals	EPA Region 1 Tier III data validation. Criteria for SW-846 6010B/7471A listed in Worksheets #12, #15, #24, and #28. If not included in Worksheet #12, #15, #24, or #28, the logic outlined in Region 1, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, modified February 1989, will be used to apply qualifiers to data.	Tetra Tech Data Validation Coordinator (Joseph Samchuck) and staff chemists

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

- **Completeness:** For each matrix that was scheduled to be sampled, the FOL acting on behalf of the project team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

- **Precision:** The Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (*J qualifier), limitations on the use of the data will be described in the project report.

- **Accuracy:** The Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

- **Representativeness:** A project scientist identified by the Tetra Tech PM and acting on behalf of the project team will determine whether the data are adequately representativeness of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, be reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

- **Comparability:** The Project Chemist acting on behalf of the project team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Project Chemist indicates that such quantitative analysis is required.

- **Sensitivity:** The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

Data Usability Assessment

- **Project Assumptions and Data Outliers**
  The Tetra Tech Project Manager and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumpt9ions include assumptions related to data distributions (e.g., Normal versus log-normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. potential outliers will be removed if a review of the associated indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the project manager will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be sued to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

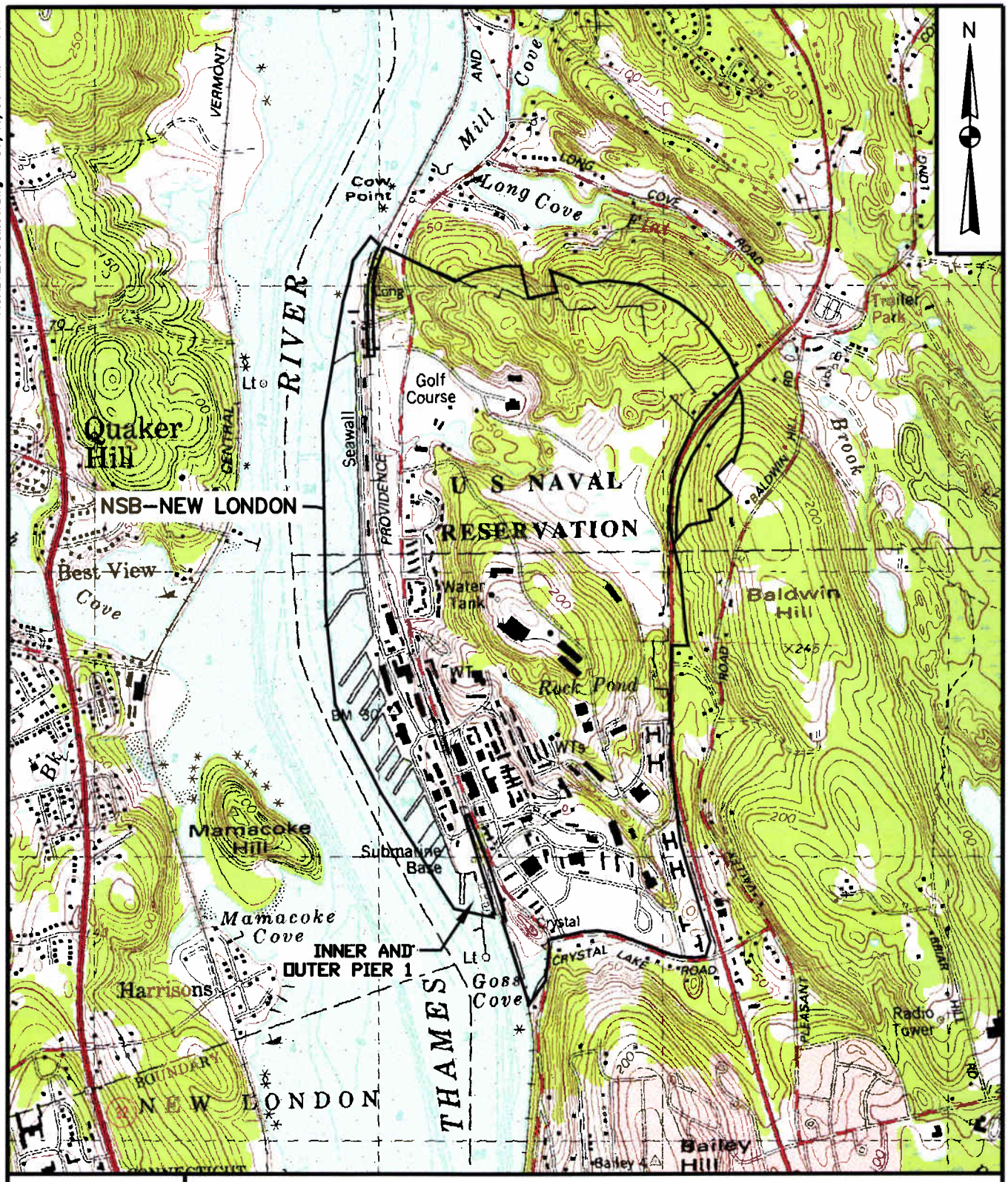
The Tetra Tech Project Manager, Project Chemist, FOL, and project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, NSB-NLON Environmental Restoration Program Manager, EPA RPM, and the State of Connecticut Project Manager. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simple be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

FIGURES

ACAD: 2195CM01.dwg 06/11/09 MF PIT



0 2000 4000
GRAPHIC SCALE IN FEET

SOURCE: USGS QUADRANGLE MAP UNCASVILLE, CONNECTICUT, 1984

DRAWN BY MF	DATE 6/11/09	Tetra Tech NUS, Inc.	CONTRACT NO. 2195
CHECKED BY	DATE	LOCATION MAP INNER AND OUTER PIER 1 NSB-NLON, GROTON, CONNECTICUT	OWNER NO. WE24
REVISED BY	DATE		APPROVED BY CAR DATE 6/16/09
SCALE AS NOTED			DRAWING NO. FIGURE 10-1 REV. 0

FORM CADD NO. TTNUS-AV.DWG - REV 1 -9/10/98

P:\GIS\NLON\MAPDOCS\MXD\PIER_1.MXD 8/31/09 TW

*** SBP-1 - Accuracy of historical data point not able to be validated**

Sample ID	Northing (NAD 83)	Easting (NAD 83)
Existing		
P1	703756.322	1181222.215
P1-C1	703808.871	1181223.09
P1-48	703732.676	1181234.476
SBP-1	703818.505	1181260.75
Proposed		
TRP1-SD-011	703822.333	1181222.657
TRP1-SD-012	703792.013	1181199.918
TRP1-SD-013	703793.273	1181249.197
TRP1-SD-014	703808.633	1181230.237
TRP1-SD-015	703816.023	1181254.247
TRP1-SD-016	703755.372	1181230.238
TRP1-SD-017	703730.832	1181240.688

DRAWN BY T. WHEATON	DATE 8/31/09	Tetra Tech NUS, Inc.	CONTRACT NO. WE24 / OWNER NO. 2195
CHECKED BY C. RICH	DATE 8/31/09		APPROVED BY / DATE
COST/SCHEDULE AREA		STUDY AREA AND SITE LOCATION MAP INNER PIER 1 AREA NSB-NLON, GROTON, CONNECTICUT	APPROVED BY CAR / DATE 9/2/09
SCALE AS NOTED			FIGURE NO. 10-2 / REV. 0

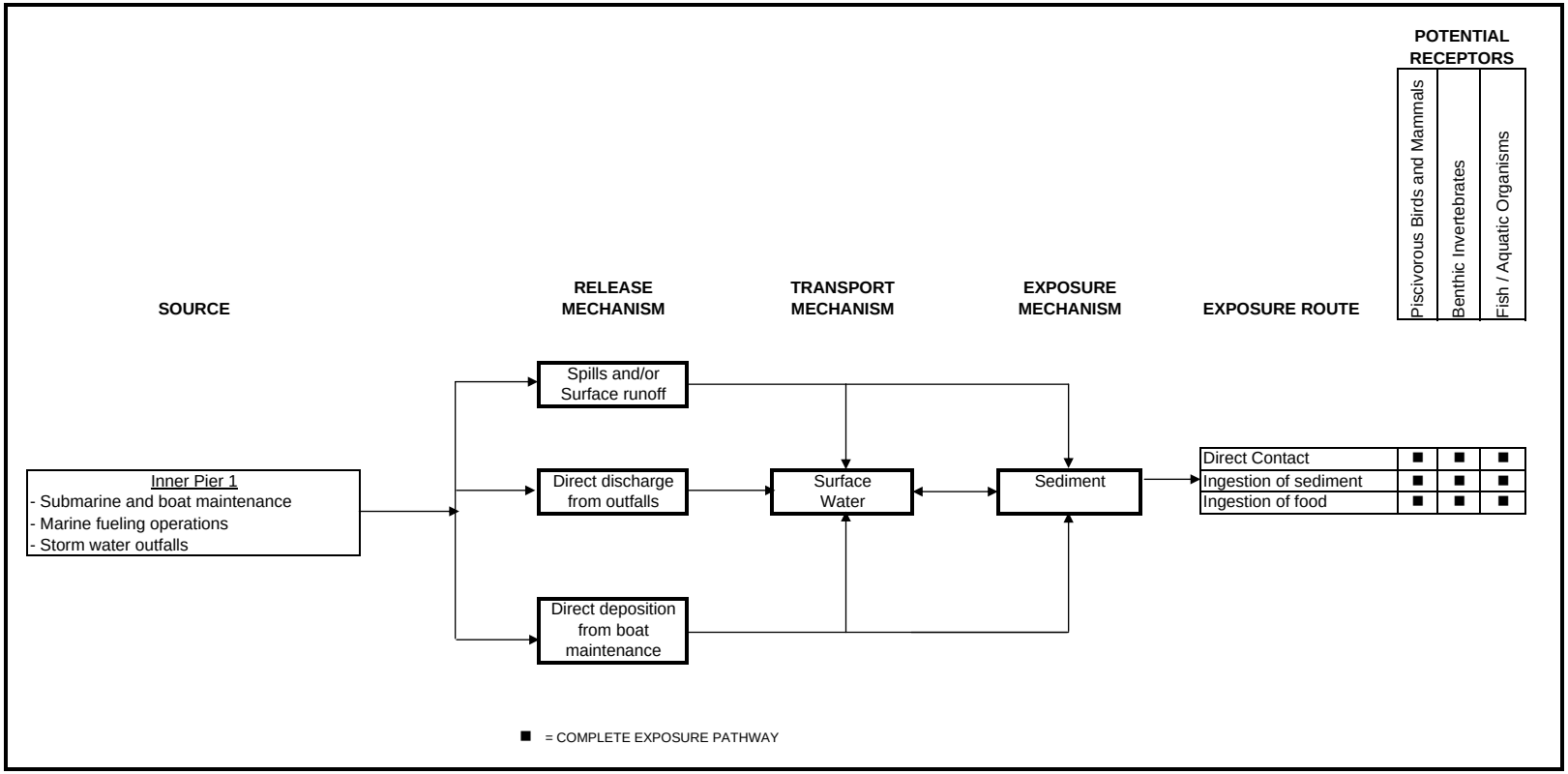
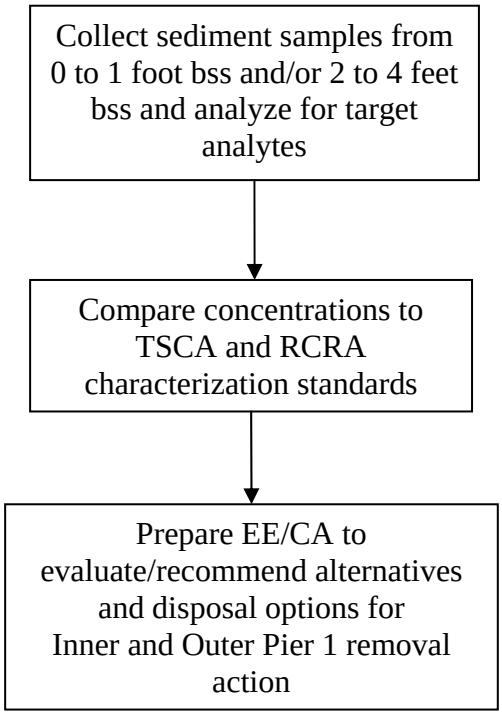


FIGURE 10-3 ECOLOGICAL CONCEPTUAL SITE MODEL
THAMES RIVER - INNER PIER 1
NSB - NLON, GROTON, CONNECTICUT

Figure 11-1. Inner Pier 1 Study Goals



APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-6.1	Page 1 of 11
		Effective Date 02/04	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory <u>before</u> sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed <u>prior to</u> the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

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U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

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ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration		Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER					
Organics (GC&GC/MS)	VOC Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables (Low SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
	Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	14 days
SOIL					
Organics (GC&GC/MS)	VOC	EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables (Low SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium	Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All	Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All	Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR					
Volatile Organics	Low/Medium	Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2[9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5[10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field

technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

Bridge/Boat Sampling – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United states Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.

- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.

2. Inexpensive cost (if the item is to be disposed).

3. Ease of operation.

4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

Safety Reminder

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.

2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.

3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.

4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.

5. Use a clean paper towel to clean and dry the outside of the container.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects

sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.
3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
4. Raise the sampler and cap the bottle.
5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.
2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.
3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).
4. Use a paper towel to clean and dry the outside of the container.
5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.

- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.

- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.

- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.

- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be

collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices
- A minimum of three people are required if *any* of the following conditions are anticipated or observed:
  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.
  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.
  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.

2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.
2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.
3. Open the sampler to retrieve the sediment.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.
6. Use a paper towel to clean and dry the outside of each container.
7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

SAFETY REMINDER

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices. All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

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Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1984. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-84-017.

U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.

ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA

	Color Visual	pH Standard	S.C. mS/cm	Temp. Degrees C	Turbidity NTU	DO mg/l	Salinity %	Other NA
Date:								
Time:								
Depth:								
Method:								

SAMPLE REQUIREMENT INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS/NOTES | **MAP**

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA

Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA

Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES

MAP

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 Surface Water Sample Collection

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be plased in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	Project: Site: Location:	
Sample No:		Matrix:
Date:	Time:	Preserve:
Analysis:		
Sampled by:	Laboratory:	

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR ___	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**Signature**
Date	**Date**
Signature	**CUSTODY SEAL**

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - Is a solution selected/identified within the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

Potable Water - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

Solvent - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Failure to meet these objectives are sufficient to restrict equipment from entering the site/exiting the site/ or moving to a new location on the site until the objectives are successfully completed.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide the following characteristics

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

  - Liner – Depending on the types of equipment and the decontamination method the liner should be of sufficient thickness to provide a puncture resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. Achieving the desired thickness maybe achieved through layering lighter constructed materials. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner a light coating of sand maybe applied to provide traction as necessary.

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.

2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

- The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

1) Wash with soap and water
2) Rinse with tap water
3) Rinse with deionized water

Note: In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer's instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.

- Cleanliness – As per protocol only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or associated with noticeable odors it should be decontaminated prior to use.

  1) Wash with soap and water
  2) Rinse with tap water
  3) Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole Drilling Equipment

This includes any portion of the drill rig that is over the borehole including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

During the course of field investigations disposable/single use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- Rinsate Blanks – It is recommended that Rinsate samples be collected to

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

001 - Monitoring Well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

5.5 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature)

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site specific planning documents.

TG&B Vibrocoring Procedure

1.0 Safety

1.1 Before working, check all boat rigging, cables, shackles and cotter pins.

1.2 When getting to the work site, raise the 'ball-diamond-ball' shapes and announce your activity, location and duration on marine channel 13.

2.0 Getting on Station

2.1 Position the boat on the approximate core location, determine whether the wind or current is dominant (setting direction). Note that all positioning is dependant on vessel traffic and the channel orientation.

2.2 Run the boat approximately 100 feet up wind (or current) and set the bow anchor.

2.3 Back down to the core location. When on station, lower spuds. If the water depth is too deep for spudding, set 2 anchors (one off each stern quarter) out approximately 100 feet.

2.4 Insure anchor lines are tight and the boat is not drifting.

3.0 Core Preparation

3.1 Measure and log water depth with weighted tape. Refer to tide table for tide correction. Calculate MLW water depth and record it.

3.2 Cut core tube to 1.5 inches longer than core pipe and deburr ends.

3.3 Thread plunger cable through tube, attach plunger and set it 4-5 inches inside tube.

3.4 Insert retaining fingers in lower end and insert core tube in core pipe. Check bottom end for proper seating.

3.5 Mark on core pipe (or topping pipe if deeper) the measured water depth. Measure and mark on topping pipe distance to required penetration.

4.0 Coring Operation

4.1 Review responsibilities between captain and coring engineer. Put on nitrile gloves and hearing protection.

4.2 Start air compressor.

4.3 Raise the vibrator head to shoulder level and bolt in core pipe.

4.4 Insure that the plunger wire is properly fed and clear.

4.5 Lower coring assembly so that the depth mark is at the waterline.

4.6 Secure (cleat) plunger cable.

4.7 Simultaneously slack off the main winch wire and open the valve to introduce air to the vibrator.

4.8 Continue coring operation until the required penetration depth is attained or progress is stopped (refusal).

4.9 Record penetration depth.

4.10 Using the main winch wire, retrieve coring assembly. Raise it to approximately head level.

4.11 Uncleat the plunger wire.

4.12 Put a stopper line on the core pipe and unbolt the core pipe from the vibrator.
4.13 Remove the core pipe, taking the top end forward.
4.14 Carefully remove the core barrel, cap and tape both ends.
4.15 Measure recovery and log it.
4.16 Mark station number, top end, penetration and recovery data on the tube.
4.17 Stand core up vertically and secure it.

5.0 Deconn

5.1 Deconn core pipe end, plunger and fingers in preparation for next station.

APPENDIX B

FIELD FORMS

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

PROJECT NAME: _____ **PROJECT NUMBER:** _____

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

Tt NUS PERSONNEL: _____ **DEPARTURE TIME:** _____

CONTRACTOR: _____ **DRILLER:** _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

_____

_____

_____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

Tetra Tech NUS, Inc.

PROJECT: _____ JOB #: _____
LOCATION: _____ DATE: _____
PROJECT MANAGER: _____ FOL: _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1			
2			
3			
4			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

APPENDIX C

LABORATORY TECHNICAL SPECIFICATION AND CERTIFICATION LETTER

Sharyn Lawler [Mitkem]

From: Moreno, Pati (NFESC) [pati.moreno@navy.mil]
Sent: Monday, March 09, 2009 8:03 PM
To: Sharyn Lawler [Mitkem]
Subject: Navy Assessment - Mitkem
Signed By: There are problems with the signature. Click the signature button for details.

Ms. Lawler,

This email addresses the status of Mitkem Laboratories located at 175 Metro Center Boulevard, Warwick, Rhode Island in the Navy Environmental Restoration (ER) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

NAVSEA 04XQ conducted a laboratory assessment of Mitkem Laboratories located in Warwick, Rhode Island as a support service to the Naval Facilities Engineering Service Center (NFESC). The general conclusion of the assessment is that the laboratory has successfully completed the evaluation for the parameters summarized in NAVSEA 04XQ letter Ser 04XQ (LABS)/095 dated May 7, 2008, and the supplemental EPA Method 8015 review; these results are applicable to the Navy ER QA Program administered by NFESC. Based on the outcome of the assessment, a re-evaluation of your laboratory under the Navy ER QA Program will be due not later than March 11, 2010.

The outcome of this assessment does not guarantee the delivery of any analytical samples, and is facility specific (i.e. is not applicable to an affiliated or subcontract laboratory). The Navy reserves the right to conduct additional laboratory assessments. The outcome of the assessment may result in the addition or removal of parameters listed in the original scope of review, or reclassification from successful to unsuccessful.

Please contact me if there are parameters not presented on the table in the aforementioned NAVSEA letter that the laboratory expects to run on a routine basis in support of Navy environmental restoration projects. In these circumstances the laboratory's capability to run the tests will be assessed and the table will be modified accordingly. Any other questions concerning the information provided should also be directed to me.

Sincerely,
Pati Moreno
NFESC ER QA Program Coordinator
PH: (805) 982-1659 Fax: (805) 982-4304
Email: pati.moreno@navy.mil

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
NAVAL SUBMARINE BASE (NSB), NEW LONDON, CT

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT *N62470-08-D-1001,* CONTRACT TASK ORDER (CTO) NO. WE24

PRE-DESIGN ACTIVITIES
CHEMICAL ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract ***N62470-08-D-1001*** is procuring laboratory analytical services for NSB New London, CT in support of Pre-Design Activities. Requested analyses include Polychlorinated Biphenyl (PCB) Organic Compounds and Toxicity Characteristic Leaching Procedure (TCLP) Metals.

The laboratory performing these analyses must be certified in the state of Connecticut. Additionally the laboratory must provide a copy of their NFESC approval letter, all methods requested must be addressed.

The responding laboratory must submit method detection limits (MDLs) and quantitation limits (QLs) for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response. After award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete tables for inclusion in the Sampling and Analysis Plan (SAP). The SAP will be prepared according to the Uniform Federal Policy (UFP) for QAPPs (March 2005) and utilize the UFP SAP worksheets 1 through 37.

2.0 SAMPLE INFORMATION

Samples will be shipped to the laboratory during the period beginning Fall 2009.

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in the attached Table 1.

The laboratory must determine the percent moisture content of the sediment samples before any sample analysis, and the sample aliquot must be increased to compensate for the moisture content of the sample. The required reporting limits MUST be achieved, regardless the moisture content of the sediment samples.

Field duplicate samples will be submitted with "blinded" identification to the laboratory. The field crew will designate samples (one per twenty samples of like matrices) upon which matrix spike/matrix spike duplicate (organic analyses) and matrix spike/laboratory duplicate (inorganic analyses) are to be performed; additional volumes of these samples will be provided as necessary.

All samples are expected to be of moderate to high contaminant concentrations. The field crew will attempt to identify any potentially high concentration samples.

All positive identifications for PCBs by gas chromatography (GC with electron capture detector (ECD) analyses MUST be confirmed on a second column that possesses retention characteristics different from those exhibited by the primary column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the reporting limit but greater than the MDL must be reported by the laboratory, the laboratory must “J” flag these results. As per

the DOD QSM, the higher of the two results generated from dual column analyses must be reported as the primary result by the laboratory unless RPDs between columns are greater than 40% and interference is found to be present or there is some other justifiable reason to select the lower of the two results.

For the ICP and mercury metal analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (serial dilution) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

3.0 ANALYSIS/REPORTING INFORMATION

Data package deliverables must be submitted as a hardcopy and PDF (2 copies/2 CDs). The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Any analysis and reporting requirements addressed in the DOD Quality Systems Manual (January 2006 or April 2009 depending on current NFESC approval letter) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF data packages for PCBs and metals analysis must meet Contract Laboratory Program (CLP) format, reporting, and PDF data package deliverable requirements. The PDF data packages for all the remaining analyses must be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

Additionally, each PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP Forms 1 through 15 and non-CLP it shall be the CLP-like equivalent of Forms 1 through 15).

Attachment A details the required target compound list and required QL goals that must be met. The laboratory must submit its MDLs and QLs for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response.

Attachment B details the required summary forms for CLP-like data packages, the requirements for the organization, and bookmarking of PDF data packages.

Non-detected organic and metals results must be reported down to the MDLs submitted by the laboratory in Attachment A with the bid response. Positive results above the MDL but below the laboratory's QLs must be reported as estimated values qualified with a "J" **for both organic and metals** analyses. Soil samples must be reported on a dry-weight basis.

The PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported. All soil and sediment matrix sample results shall be reported on an adjusted dry-weight basis. Additionally, the data package deliverable must include the ORIGINAL chain-of-custody form (received with the samples and signed by the laboratory sample custodian).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of collection. The holding times are as follows:

Analyses	Holding Time
PCBs	14 days until extraction (solids); 40 days to analysis
TAL Metals	6 months to analysis except mercury which is 28 days to analysis

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, the holding time criteria apply to any/all subsequent sample dilutions and re-analyses.

The TtNUS Project Manager for this project is Mr. Corey Rich and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication it is required that the laboratory's appointed project manager contact Mr. Rich once a week for the entire project duration.

Contact information for Mr. Rich is as follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8984
Fax: 412-921-4040
e-mail: corey.rich@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All PDF analytical data package (2 CDs) and associated electronic (TXT) deliverables are due to the TtNUS Sample Management Coordinator, Ms. Tobrena Skeen, within the expedited MSA turnaround term of 14 calendar days from receipt of the last sample in a Sample Delivery Group (SDG). Additionally all SDGs must contain 20 samples. The frequency in which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed. **Additionally the laboratory Project Manager must fax copies of chain of custody forms to Ms. Skeen as samples are received by the laboratory.**

Contact information for Ms. Skeen follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8582
Fax: 412-921-4040

e-mail: tobrena.skeen@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48-hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. Samples will be shipped to the laboratory the period **beginning Fall 2009**.

Shipment of bottleware to support the entire project will be coordinated at a later date.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. **The laboratory must also provide any extra coolers needed for return shipment of sample to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms. The laboratory is to dispatch proper bottleware for the collection of additional soil, per sampling location, for dry-weight determination.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within a laboratory, internal transfers of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must maintain documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact sample aliquots, as stipulated in the MSA. **Additionally, the laboratory must store hardcopy and/or PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Kelly Carper at 412-921-7273 or via e-mail kelly.carper@tetratech.com. Ms. Carper must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Questions regarding electronic diskette deliverable concerns are to be directed to Ms. Amy Thomson at 412-921-8182 or via e-mail amy.thomson@tetratech.com. Ms. Thomson must be contacted and informed of any difficulties encountered preparing the required electronic deliverables.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte detection limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. **All costing information must reflect the terms and conditions established by the 2009 CLEAN MSA.**

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
NAVAL SUBMARINE BASE (NSB), NEW LONDON, CT
CTO WE24, PRE-DESIGN ACTIVITIES

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Solid	TCL PCB Organic Compounds	SW-846 8082 or 8082A	6	$	$
	TCLP Metals	SW-846 1311/6010B or 6010C and SW-846 7470A	7	$	$

TOTAL COST $

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there an additional charge and what is that charge?_____
Name of Laboratory_____

Signature_____

ATTACHMENT A
Required Target Compound Lists and Quantitation Limits

Matrix: Sediments
Analytical Group: PCB Aroclors

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/kg)	Mitkem[2,3]	
					Quantitation Limit (µg/kg)	Method Detection Limit (µg/kg)
Aroclor-1016	12674-11-2	1000	TSCA	333		
Aroclor-1221	11104-28-2	1000	TSCA	333		
Aroclor-1232	11141-16-5	1000	TSCA	333		
Aroclor-1242	53469-21-9	1000	TSCA	333		
Aroclor-1248	12672-29-6	1000	TSCA	333		
Aroclor-1254	11097-69-1	1000	TSCA	333		
Aroclor-1260	11096-82-5	1000	TSCA	333		

1 TSCA 40 CFR Part 761.
2 Mitkem-specific MDLs and QLs are presented.
3 QL/MDL values are based on 100 percent solid samples.

Matrix: Aqueous leachate
Analytical Group: Metals

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (µg/L)	Mitkem[2]	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
Silver	7440-22-4	5000	40 CFR 260.11	1670		
Chromium	7440-47-3	5000	40 CFR 260.11	1670		
Cadmium	7440-43-9	1000	40 CFR 260.11	333		
Lead	7439-92-1	5000	40 CFR 260.11	1670		
Barium	7440-39-3	100000	40 CFR 260.11	3300		
Arsenic	7440-38-2	5000	40 CFR 260.11	1670		
Selenium	7782-49-2	1000	40 CFR 260.11	333		
Mercury	7439-97-6	200	40 CFR 260.11	66		

1 40 CFR Part 260.11
2 Mitkem-specific MDLs and QLs are presented.

ATTACHMENT B
Summary Form Requirements for PDF Deliverable and PDF Data Package Deliverables

PDF DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide 2 compact disks (CDs) containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, General Chemistry, etc.)
a. QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
c. Calibration Data (includes all initial and continuing calibrations)
d. Miscellaneous (includes extraction forms, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6010 series for metals:

Results Report - must present all information presented on CLP FORM I.
Initial and Continuing Calibration Summary -must present all information presented on CLP FORM 2A.
CRDL Summary - must present all information presented on CLP FORM 2B.
Blanks - must present all information presented on CLP FORM 3.
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4.
Matrix Spike Summary - must present all information presented on CLP FORM 5A.
Post Digestion Spike - must present all information presented on CLP FORM 5B.
Lab Duplicate Results - must present all information presented on CLP FORM 6.
LCS Summary - must present all information presented on CLP FORM 7.
MSA Summary (Method of Standard Addition) - must present all information presented on CLP FORM 8.
ICP Serial Dilution Summary - must present all information presented on CLP FORM 9.
Detection Limits - must present all information presented on CLP FORM 10.
Linear range – must present all information presented on CLP FORM 12
Prep Log - must present all information presented on CLP FORM 13.
Analysis Run Log - must present all information presented on CLP FORM 14.
ICP-MS Tune – must present all information on CLP FORM 14 (ILM05.4)
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

<u>Also must include</u>: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

Summary Forms for SW-846 8082 Polychlorinated Biphenyl (PCB) Organic Compounds (all other SW-846 GC methods) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Pesticide Initial Calibration of Single Component Analytes	Present all information contained on CLP Form VI-PEST-2.
Summary of Pesticide Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Pesticide Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Pesticide Identification Summary For Single Component Analytes and for Multiple Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com.

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.	Text	25	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte	Text	12	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.			
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, **do not enter "ND" or "0". Do not leave this field blank**. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0"	Numeric (1)	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard	Text	7	Q

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	values table may be used. If so, these must be added to the standard value table in the application.			
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or surrogate was not recovered because it was not added to the sample, enter “NS”.	Numeric (1)	5(3)	R
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	D
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	the project library or QAPP.			
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount.	Numeric (1)	10(6)	R
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	R
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
R Only required if Analyte_Type =”SPK” or “SURR”.
D Only required for spiked compounds in the LCSD and MSD.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD	Date/ Time	16*	C
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No	Text	25	C

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	value is required for method blanks, LCS, and LCSD.			
Temperature	Temperature (in centigrade degrees) of the sample as received. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	C
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	For radiochemistry leave this field blank. For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter “WET” if results are not corrected for	Text	3	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	percent moisture. Enter "DRY" if percent moisture correction is applied to results.			
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank.	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Leave this field blank if the sample analysis was not performed on a leachate.	Text	10	L
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Leave this field blank if the sample analysis was not performed on a leachate	Text	12	L
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. Leave this field blank if the sample analysis was not performed on a leachate	Date /Time	16*	L
Percent_Moisture	Percent of sample composed of water. Enter for soil and sediment samples only.	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.			
RunBatch	For radiochemistry methods leave this field blank. For all other methods the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. This field is only required if the A2 table is included with the EDD.	Text	12	X
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	This field is only required if the A2 table is included with the EDD.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
L Only required if analysis performed is SPLP, TCLP or WET extracts.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the "Check Files" button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the "Check Files" button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the "Print this Page" button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

APPENDIX D

LABORATORY STANDARD OPERATING PROCEDURES

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME.

SOP 20.0003

Rev. 3

	Signature	Date
QA Director:	Sharon B Lauk	4/9/08
Lab Director:	[illegible]	4/9/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME

Rev. 3

1 Scope and/or Applications

This Standard Operating Procedure (SOP) describes the procedures used to create workorders and log samples into Omega ME. Other steps in sample receipt and chain of custody documentation are described in separate SOP, Number 30.0003, entitled SOP for Sample Receipt, Storage and Tracking

2. Personnel Qualification and Responsibilities

Employees should have read and understood this SOP.

3. Summary of Procedure

This document assumes that a client and project profile has already been defined in Omega ME.

IMPORTANT: Omega ME generates unique sequence numbers (workorder numbers). These are assigned in order as samples are received at the laboratory. **It is essential that all new workorder numbers be assigned in sequence.** This number is assigned by the laboratory and is recorded on the chain of custody form **before** the samples are logged into Omega ME. The Omega ME work order number and the Mitkem project number are the same. If care is not taken, the information logged into Omega ME Work Order and the information in the Mitkem Project folder will not match.

4. Sample Preservation, Container, Handling and Storage

See separate SOP, Number 30.0003, entitled SOP for Sample Receipt, Storage and Tracking

5. Interferences and Potential Problems

Not applicable

6. Equipment and Apparatus

Not applicable

7. Reagents

Not applicable

8. Procedure

8.1 CREATING A WORKODER

8.1.1 From the main page of Omega ME click [WorkOrders] Figure 1.

8.1.2 The Omega ME - WorkOrders Form will open.

8.1.3 On the menu bar at the top of the screen click the [Add] button. This will add the next available WorkOrder and clear the screen of any prior information.

On the screen click the down arrow next to the [Client ID] button to open a list of all of Mitkem's current clients. Instead of searching through the long list of clients type in the first letters of the client name to shorten the selection. Select the proper client. The client name and address will automatically appear in the Client Information box. Figure 2.

Choose the appropriate project by pressing the down arrow on the [Client Project] button, Figure 3. If the client provides a purchase number (PO) enter it in the box provided.

Enter the received date (date samples received by the lab). It is necessary to assign the WorkOrder numbers in order by date samples received. The hardcopy due date [HC Due] is then filled in automatically by Omega based on the number of days (TAT HC/FAX) listed.

If a workorder requires a short turnaround time **(24,48 or 72 hours, etc.)**, click the checkmark for the [**Fax Results to Client**] box. The cursor will automatically move to the **(TAT HC/FX)** box which is the box on the right-hand side in which the turnaround time number will need to be entered: **24=1,48=2,72=3**. When this is complete tab down to the box that is highlighted red which will exhibit the date the workorder is due. If the hardcopy due date differs from the fax due date, enter this information now.

For EPA workorders the client is **EPAINV.** The project (I,II,III,IV,V,VI,VII,VIII,IX,X, SOM_PE) is next selected by looking at the Scheduling Notification Form. This form identifies the case number, the Region the samples are from, the analyses required and

turnaround time. Setting up an EPA workorder differs from a commercial workorder in that the Case Number and SDG fields must be completed from the Chain of Custody after protocol has been selected. In some cases, a particular Case will have samples for both water and soils. The soil samples and aqueous samples may be logged into separate Work Orders. The Mitkem project manager will provide this information. Some EPA / commercial projects are kept open for more than one day, so be careful to change the received date as the samples arrive at the lab in order to update the due date properly. Click the [Login] button to begin logging in the samples.

8.1.4 A new screen appears in order to log in the sample information. The fields that need to be completed include:

1. **Click on ADD Samp.** This will add the first sample to be logged in or the information entered into the other fields will not be saved. **Date Received:** Enter the date and time that the samples were received, **Figure 4.**
2. **Client Sample ID:** Type the client ID for each sample as it appears on the Chain of Custody. **Tag Number:** If the ID needs to be truncated because it is more than 10 characters long, type the truncated ID in the Tag Number field. Example: **SB29S-32-12302 = 29S3212302.**
3. **Collection Date:** Enter the date and time that the sample was collected, if a date and time are present. A date is necessary as this is used to keep track of hold times (for non-EPA work). The time of collection is not a necessity, unless the analysis has a 24-hour or 48-hour hold time.
4. **Matrix:** Select the down arrow and choose the appropriate matrix for the sample. If a solid matrix is chosen the PMOIST automatically appears to schedule a percent solids analysis.
5. **Bottle Type:** Choose the type of bottle for each sample fraction by clicking the down arrow and selecting the appropriate bottle.
6. **Containers:** Enter the total number of bottles received per sample.
7. **Storage/Status:** Type the location where the bottle will be stored in the Sample Receiving area. Most of the samples are stored in the walk-in (R1). VOA samples are stored in the refrigerators in the VOA lab. Aqueous metals are stored outside the walk-in cooler on a metal rack.
8. **COC ID:** Some clients require this information to be included. The Mitkem project manager will notify the Receiving department when this needs to be done. The COC ID is required for SOM01.2. If there is more than one COC, associate the correct COC ID to the correct sample. To input the COC ID, go to the AFCEE tab and type in the COC ID in COC ID. Figure 5
9. **SA (CODE):** If the workorder requires the COC ID field to be completed, then this field also must be completed. Select whether the sample is a FB (field blank),TB (trip blank), RB (rinsate blank), or EB (equipment blank). This information can be found on the COC. This field is found under the AFCEE tab
10. **Temp (C): The temperature of the cooler needs to be completed for SOM01.2. In EPA cases where there is more than one cooler received for a particular Case, the correct temperature needs to be associated with the correct samples.** This field is also found under the AFCEE tab.

11. When all the above information has been entered, the next step is to choose the correct test. The tests for a particular project may be found by clicking on the Test column and using the drop down arrow.
12. Click ADD Frac if another fraction needs to be added. Fractions are added when different bottles with different preservatives are present. For example, a client sends in a sample for volatile, semivolatile, pesticides, metals and cyanide analysis. There will be an "A", a "B", a "C" and a "D" fraction. For example, they will be logged in as "A" for volatile, "B" for semivolatile and pesticides, "C" for metals and "D" for cyanide. This is due to each sample bottle being different and having different preservatives. Figure 6.
13. To add additional samples, click on the ADD Samp button. Enter the sample ID, date and time of collection, matrix, type of bottle and storage location. Once all this info has been entered, choose the test. An easier method when logging in a number of samples with identical analyses is to use the Multilog button, Figure 6. Clicking the Multilog button automatically copies all information to the next sample except for Client ID, Date andTime of collection, and Tag Number. If multi-logging, type the client ID, date of collection and time of collection for each sample. Once this is done, click on POST. Figure 7.
14. If samples arrive with designated QC (duplicates and matrix spikes) then check the MS button on the right side of the screen after you have chosen the appropriate test Be sure to change the number of bottles for this sample, as there will be more sample bottles. Figure 8
15. If specific tests are included on the Chain of Custody but the analysis is on hold, then check the "Sample on Hold" box. Figure 9
16. Comments should be added if warranted. For example, if there is limited sample volume, or the Chain-of-Custody indicates that the samples are dirty. Go to the Comments: and type in the necessary comment.
17. Labels are printed and affixed to the bottles. Labels may be printed by clicking on the Label button. Click over the samples in which labels need to be printed. Click the OK button and then the printer icon.
18. Once the labels have been printed, a work order needs to be printed. Back out to the work order page. Click on WO Rpt. Place the work order into the folder. Figures 10 and 11
19. A preliminary invoice will also need to be printed and placed into the folder. Click on the Reports button. Click on Base Report. Click on Clear All. Check on Invoice and then click on Preview Report and print the invoice. Figures 12, 13 and 14

8.1 5 Any correction to the workorder that greatly impacts the workorder such as a significant change in holding time or turn around time, the addition of new tests, and/or the addition or cancellation of samples must also be verbally communicated to the appropriate lab supervisor by the project manager. An email is also an effective means of communication.

8.2 REVIEW of the Work Order

8.2.1 The Mitkem Project folders are handed out by the Project Managers. It is the responsibility of the project manager to ensure that the sample ID, date and time of sample collection, date of sample receipt and the correct tests are logged in for each sample. Corrections are made, if necessary, and a new Work Order printed out and placed in the red folder.

8.2.2 When adding a workorder that is not going to be invoiced (Mitkem work, PE, etc..) or if the workorder is cancelled one of the following steps must be performed:

1)The workorder client name is changed to Mitkem.

2)All samples are designated as non-billable

3)For PE samples the characters "PE" are added to the SDG number field.

4) If the workorder is to be cancelled with no billable fractions and/or tests, the following steps should be followed:

4.1 Fill in Log Review with the date.

4.2 Click on Cancel Work Order button

4.3 The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.

5)If the workorder is to be cancelled but some fractions and/or test codes can be billed, then follow these steps:

5.1 One sample should be logged in with NO-TEST and the correct priced added.

5.2 Fill in Log Review with the date.

5.3 Click on Cancel Work Order button

5.4 The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.

5.5 An invoice can be generated.

9. Data Reduction and Calculations

Not applicable

10. Quality Assurance / Quality Control

The Project Manager reviews the work order within one day of sample receipt. Any changes or corrections are implemented at this step.

11. Data validation and Reporting

Not applicable

12. Corrective Action Procedures

Not applicable

13. Health and Safety

Not applicable

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Not applicable

Figure 1

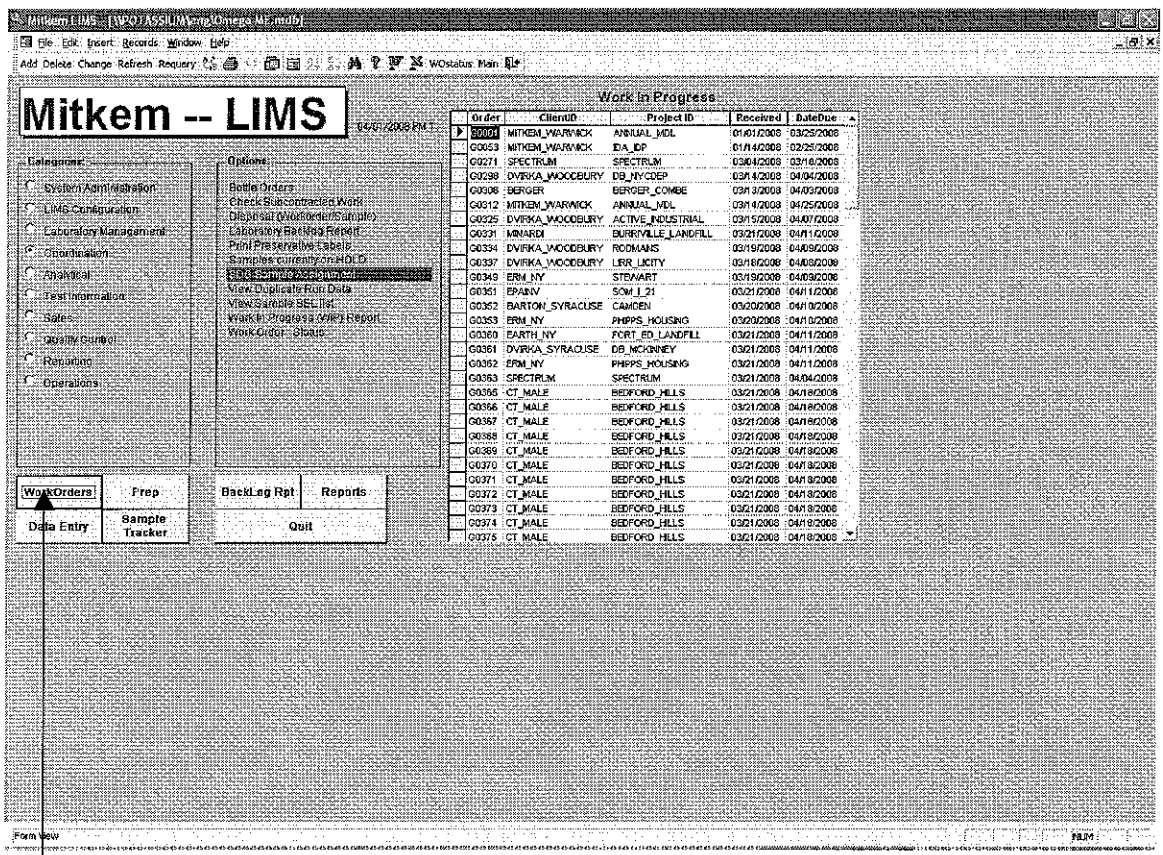


Figure 2

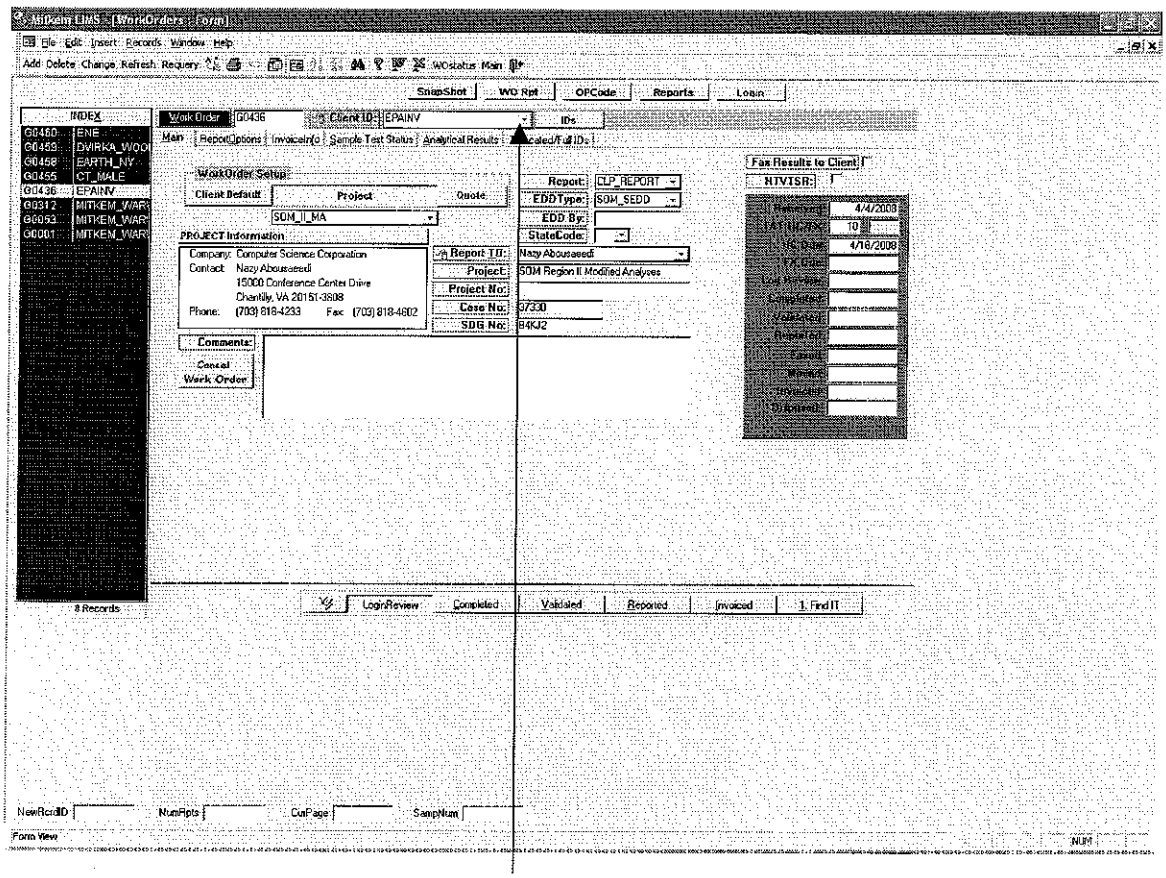

Mitkem LIMS - [WorkOrders : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0093 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order
G0436
Client ID:
EPAINV
IDs
Main
Report Options
Invoicing
Sample Test Status
Analytical Results
Scaled/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousseedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233
Fax: (703) 818-4602
Report:
CLP_REPORT
EDDType:
SOM_SEDD
EDD By:
StateCode:
Report TO:
Nazy Abousseedi
Project:
SOM Region II Modified Analyses
Project No:
Case No:
37330
SDG No:
84KJ2
Comments:
Cancel Work Order
Fax Results to Client
NTVISR:
4/4/2008
10
4/16/2008
LoginReview
Completed
Validated
Reported
Invoiced
1. Find It
NewRcdID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 3

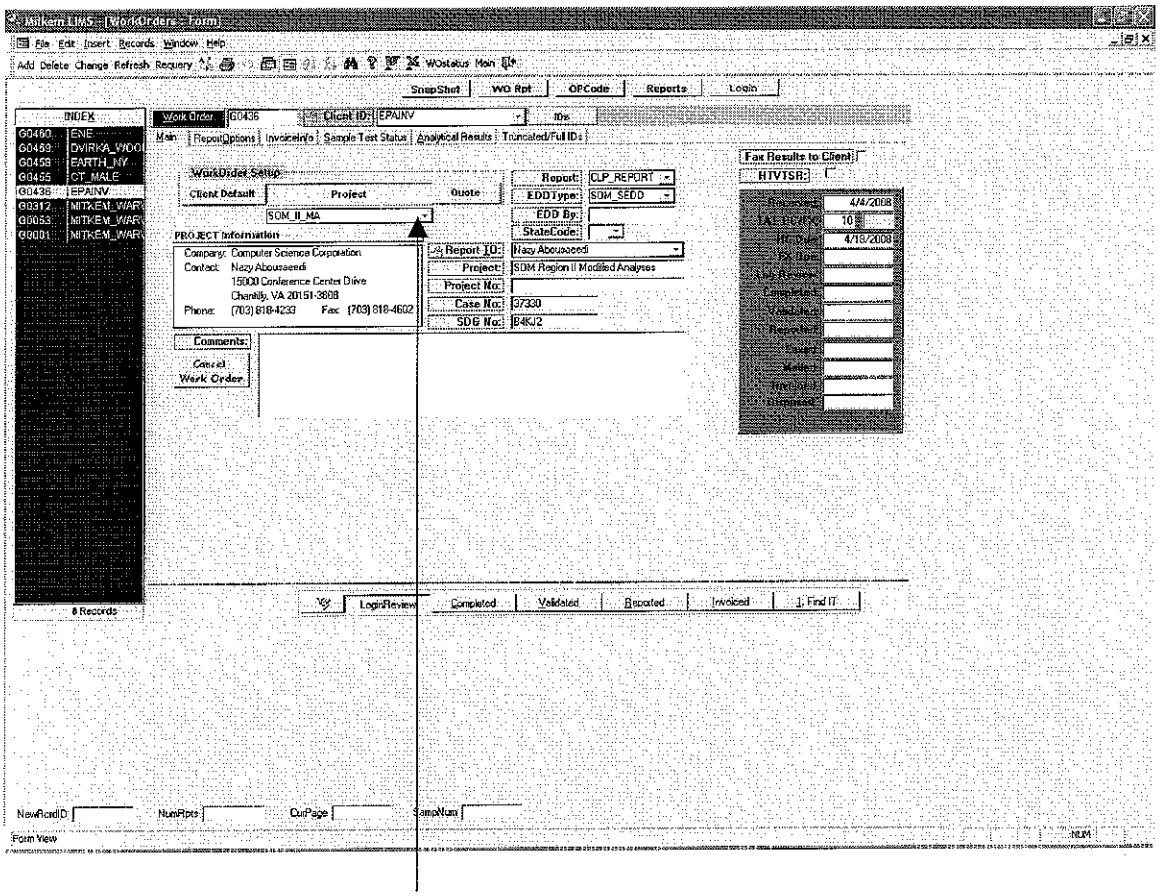

Mitkem LIMS - [WorkOrders - Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order G0436
Client ID: EPAINV
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousaeedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233 Fax: (703) 818-4602
Report: CLP_REPORT
EDDType: SOM_SEDD
EDD By:
StateCode:
Report TO: Nazy Abousaeedi
Project: SOM Region II Modified Analyses
Project No:
Case No: 37330
SDG No: B4KJ2
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
4/4/2008
10
4/18/2008
LoginReview
Completed
Validated
Reported
Invoiced
Find IT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 4

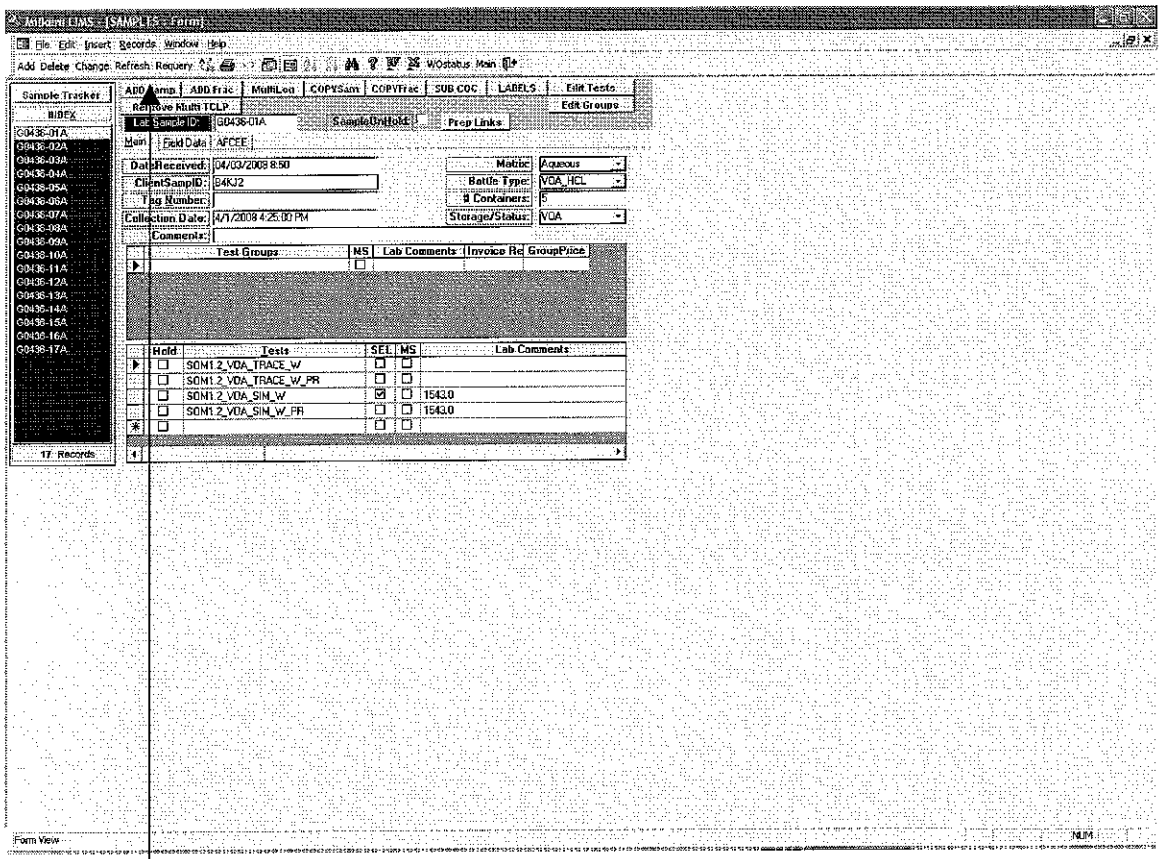

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery W/Ostatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Edit Groups
Remove Multi TCLP
Lab Sample ID:
G0436-01A
SampleOnHold
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
84KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Form View
NUM

Figure 5

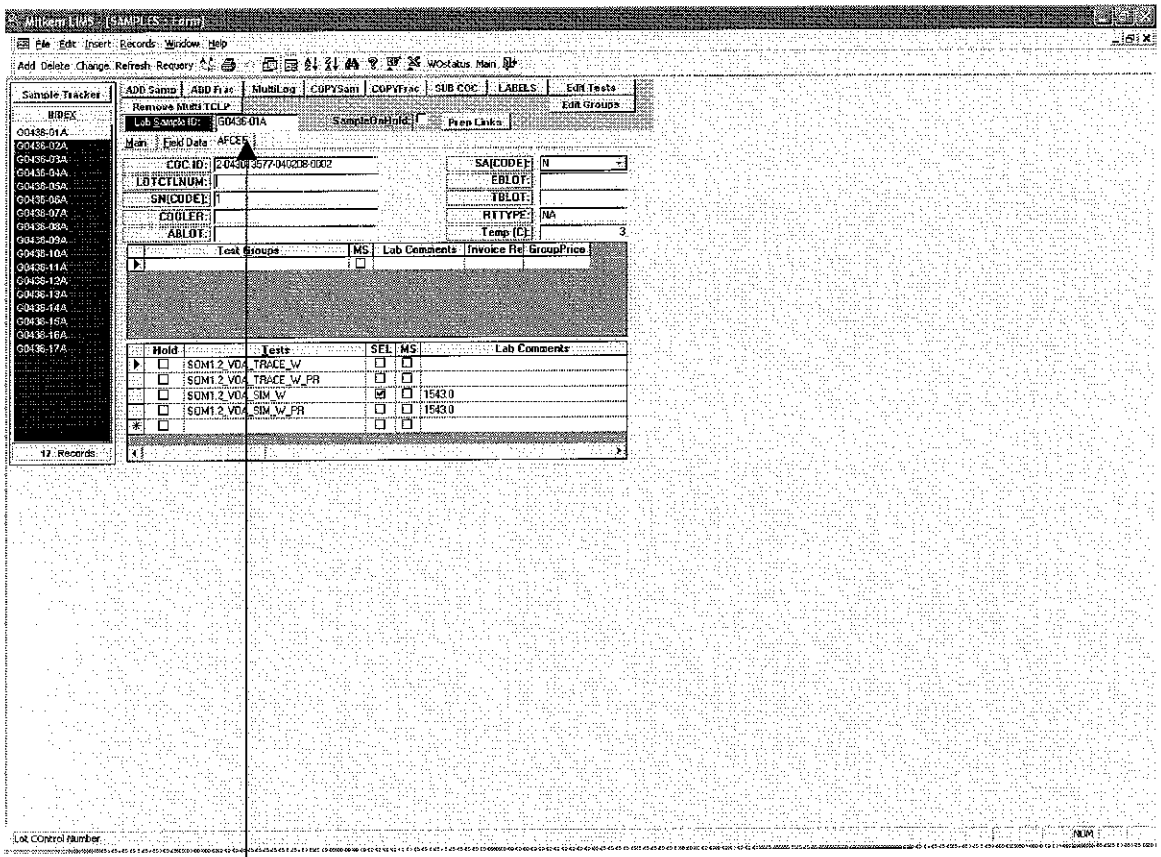

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleInstrId:
Prep Links
Main
Field Data
AFCEE
COC ID:
LOTCTLNUM:
SN(CODE):
COOLER:
ABLOT:
SA(CODE):
EBLOT:
TBLOT:
RETYPE:
Temp (C):
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
SOM1.2_VOA_SIM_W_PR
15430
15430
Lot Control Number
NUM

Figure 6

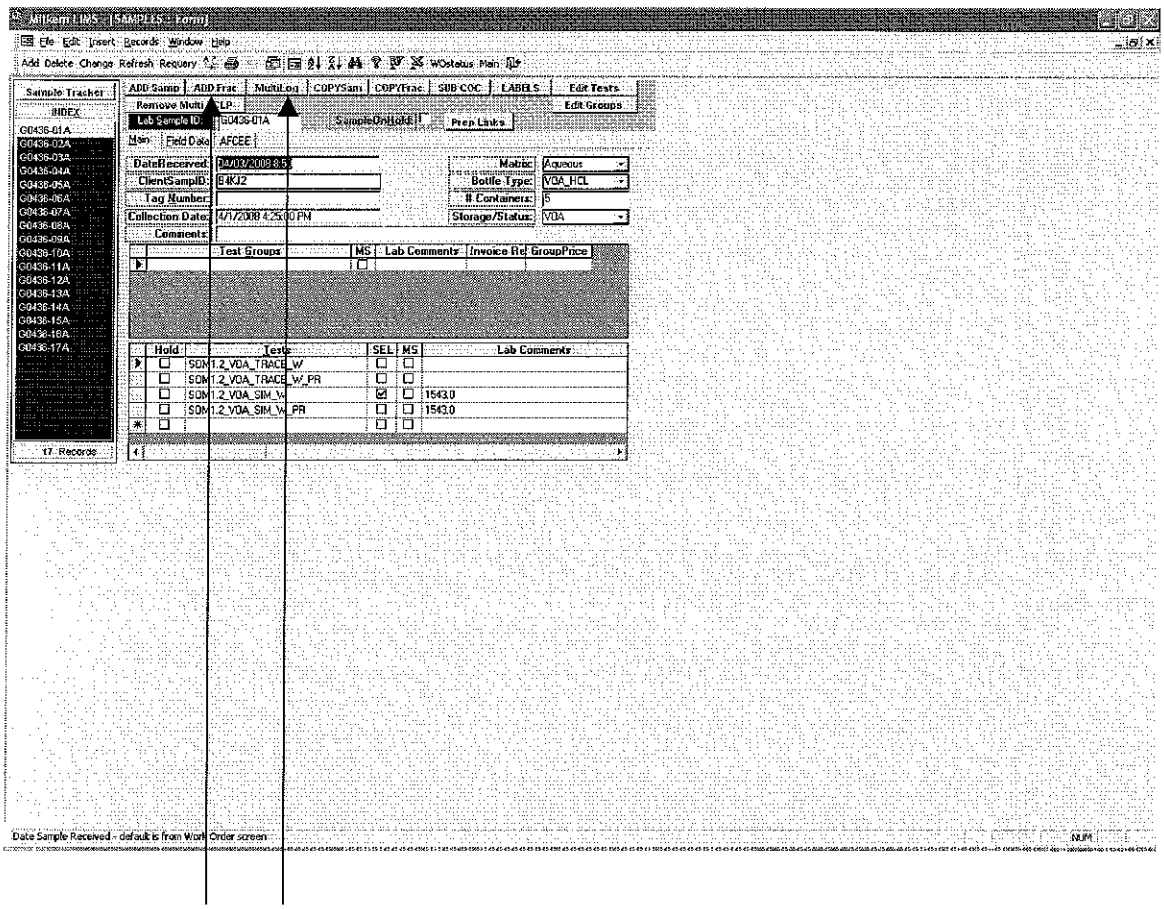

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Edit Groups
Remove Multi
Lab Sample ID:
G0436-01A
SampleOnHold
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:5
ClientSampID:
B4KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Date Sample Received - default is from Work Order screen
NUM

Figure 7

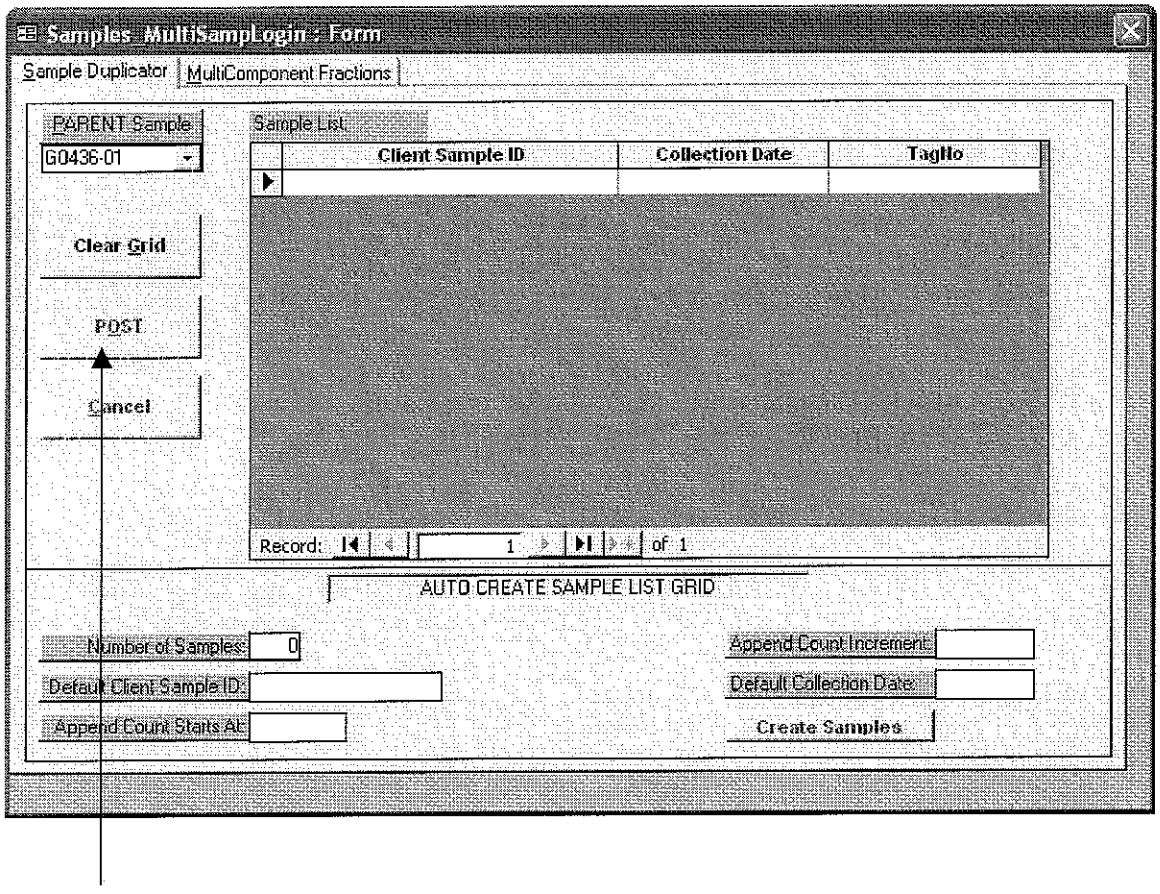

Samples_MultiSampLogin : Form
Sample Duplicator
MultiComponent Fractions
PARENT Sample
G0436-01
Sample List
Client Sample ID
Collection Date
TagNo
Clear Grid
POST
Cancel
Record:
1
of 1
AUTO CREATE SAMPLE LIST GRID
Number of Samples:
0
Default Client Sample ID:
Append Count Starts At:
Append Count Increment:
Default Collection Date:
Create Samples

Figure 8

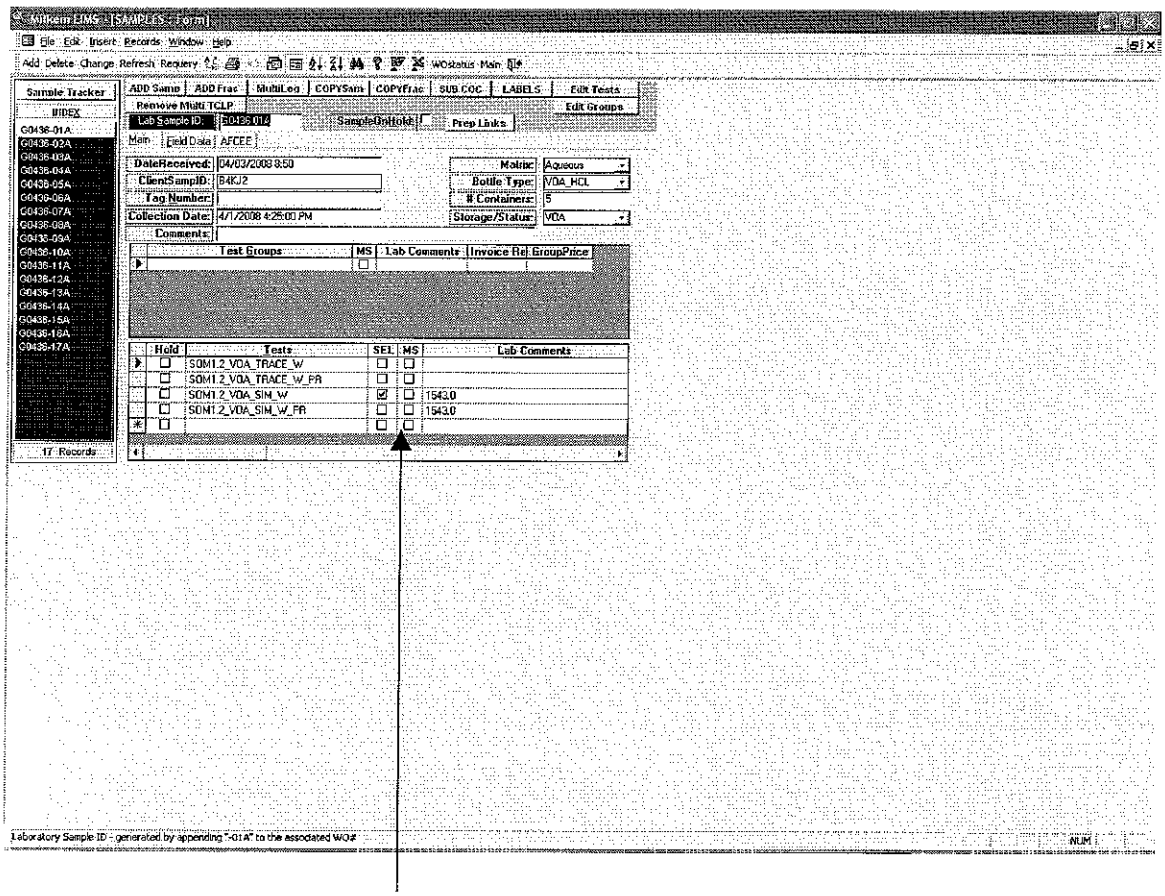

Mitkem EIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
SampleDuplicate
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
64KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 9

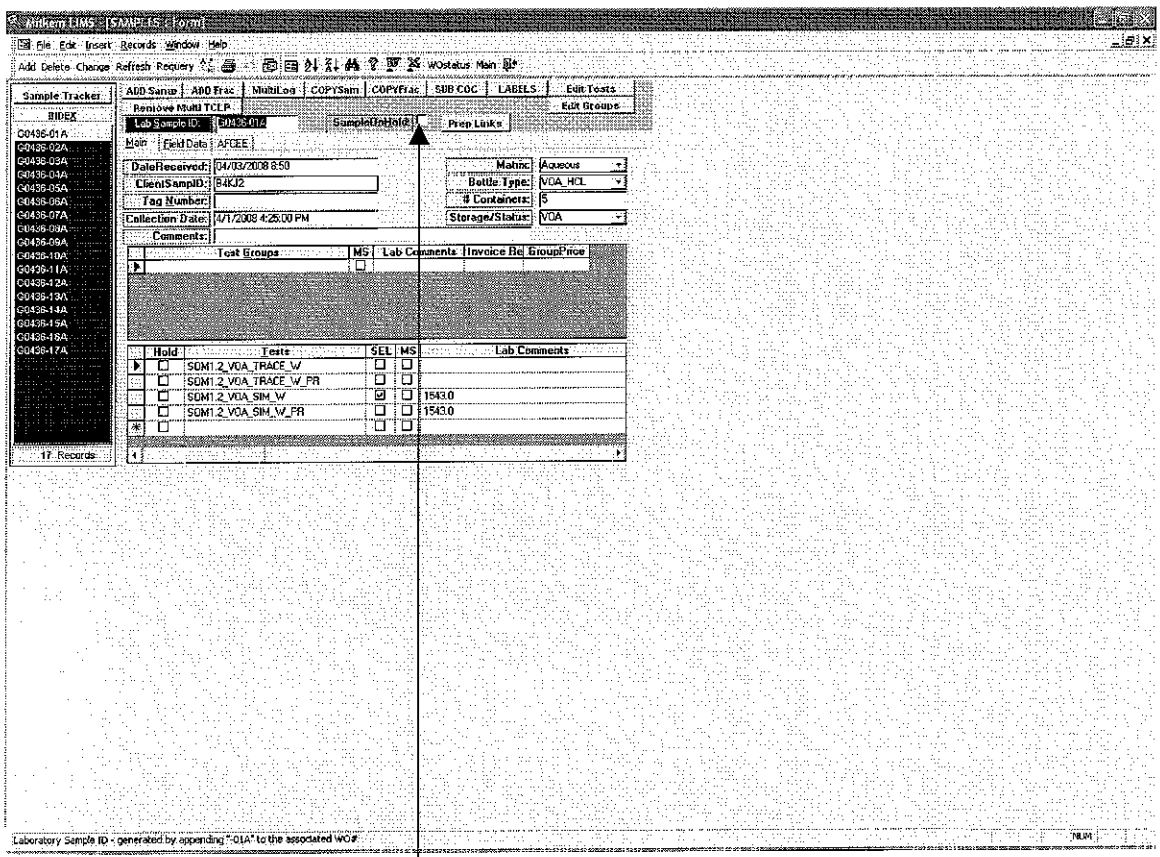

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleOnHold
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
84KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 10

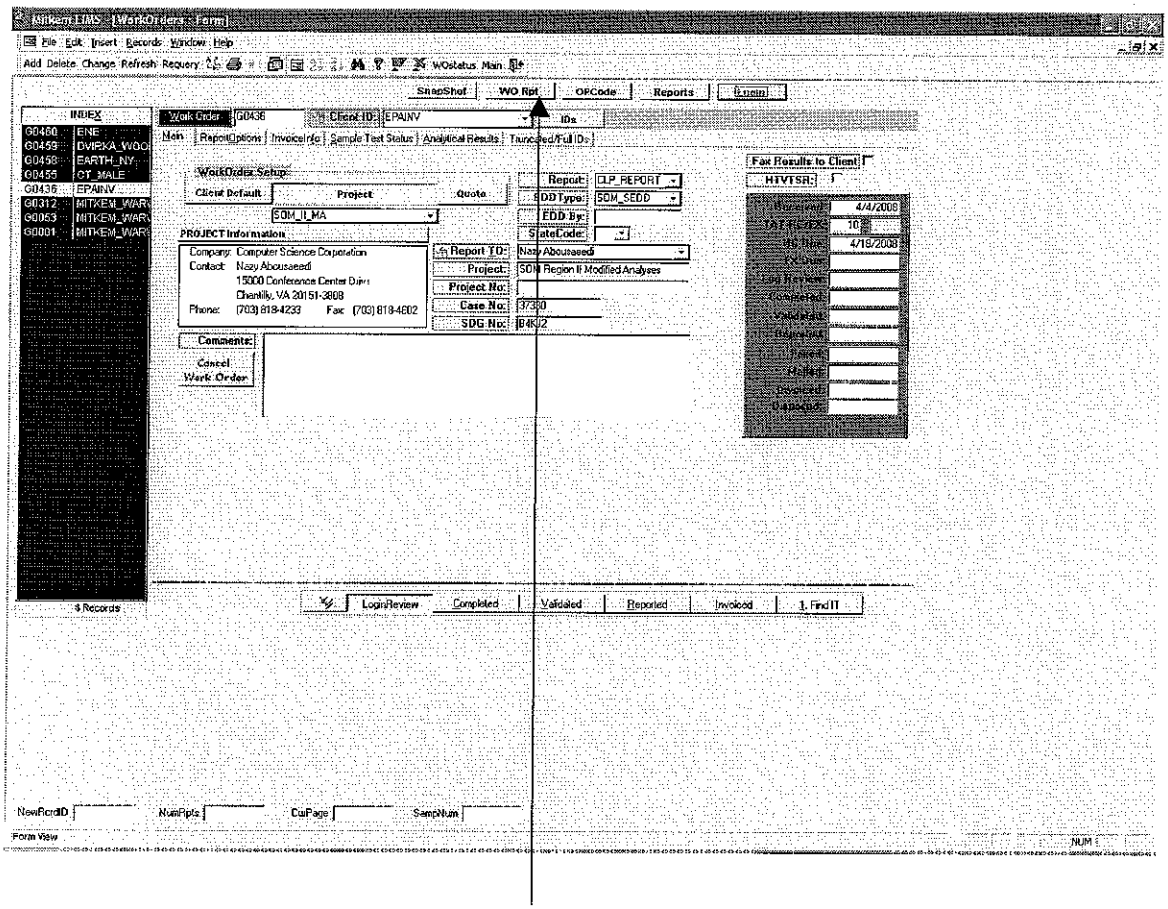

Mitkem LIMS - [WorkOrders : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
Work Order G0436
Client ID: EPAINV
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup:
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousaeedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233 Fax: (703) 818-4602
Report: CLP_REPORT
SDD Type: SOM_SEDD
EDD By:
StateCode:
Report TO: Nazy Abousaeedi
Project: SOM Region II Modified Analyses
Project No:
Case No: 37330
SDG No: B4K02
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
4/4/2008
10
4/19/2008
LoginReview
Completed
Validated
Reported
Invoiced
Find IT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 11

Mitkem Laboratories *08/Apr/08 16:19* **WorkOrder: G0436**

Client ID: EPAINV
Project: SOM Region II Modified Analyses
Location:
Comments: N/A

Case: 37330
SDG: B4KJ2
PO: EP-W-05-030

Report Level: CLP_REPORT
EDD: SOM_SEDD
HC Due: 04/18/08
Fax Due:

Sample ID	HS Client Sample ID	Collection Date	Date Rec'd	Matrix	Test Code	Lab Test Comments	Hold	MS	SEL	Storage
G0436-01A	B4KJ2	04/01/2008 16:25	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-02A	B4KJ3	04/01/2008 12:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-03A	B4KJ4	04/01/2008 16:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-04A	B4KJ5	04/01/2008 17:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-05A	B4KJ7	04/01/2008 9:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-06A	B4KK0	04/01/2008 8:00	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-07A	B4KH4	04/03/2008 11:10	04/04/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA

Client Rep. Agnes R Ng

Page 1 of 3

Figure 12

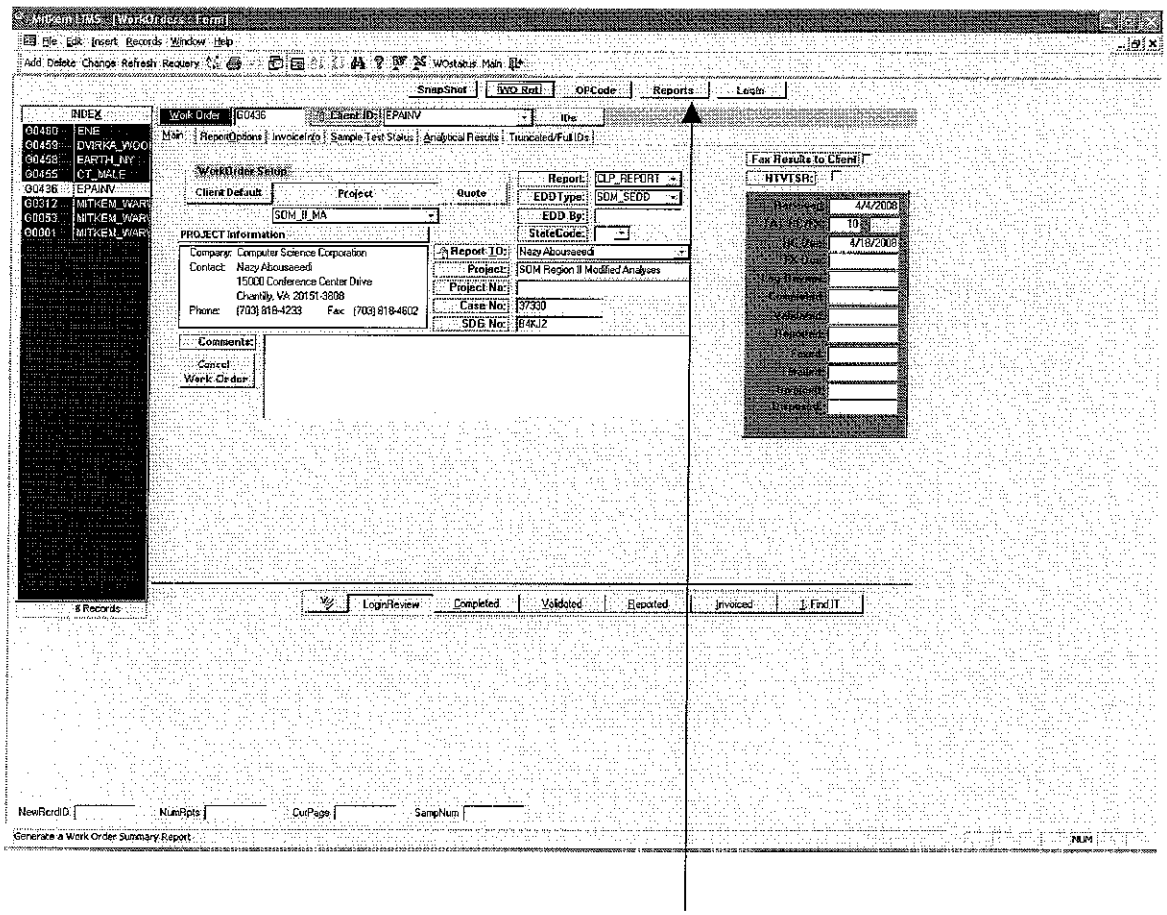

File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rptl
OPCode
Reports
Login
INDEX
Work Order
Client ID:
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup
Client Default
Project
Quote
Report:
EDD Type:
EDD By:
StateCode:
PROJECT Information
Report ID:
Project:
Project No:
Case No:
SDG No:
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
LoginReview
Completed
Validated
Reported
Invoiced
Find IT
NewRcrdID
NumRpts
CurPage
SampNum
Generate a Work Order Summary Report
NUM

Figure 13

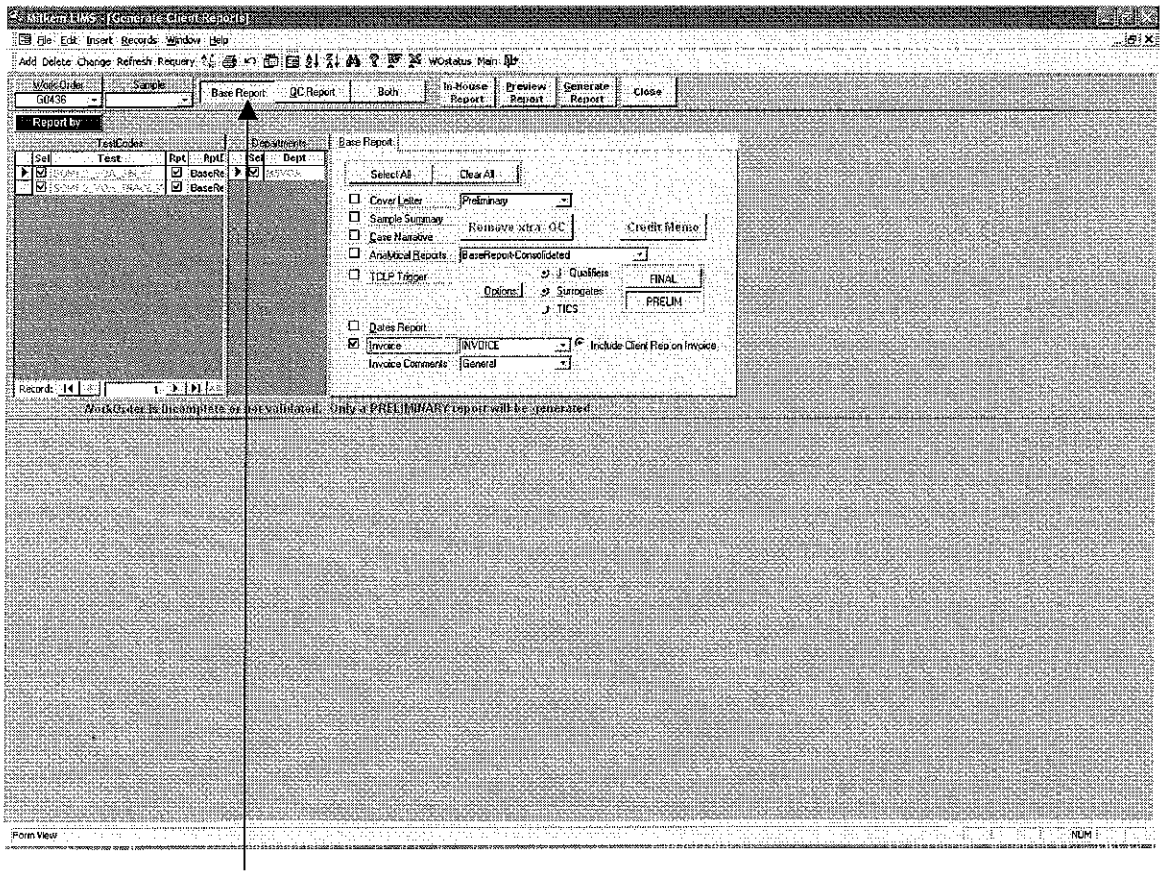

Mitkem LIMS - [Generate Client Reports]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Work Order
G0436
Sample
Base Report
QC Report
Both
In-House Report
Preview Report
Generate Report
Close
Report by
TestCodes
Departments
Base Report
Select All
Clear All
Cover Letter
Preliminary
Sample Summary
Case Narrative
Remove xtra QC
Credit Memo
Analytical Reports
BaseReport-Consolidated
TCLP Trigger
Options
Qualifiers
Surrogates
TICS
FINAL
PRELIM
Dates Report
Invoice
INVOICE
Include Client Rep on Invoice
Invoice Comments
General
Records:
Work Order is incomplete or not validated. Only a PRELIMINARY report will be generated.
Form View
NUM

Figure 14

Bill To:

INVOICE

EPA Financial Management Center
Attn:
Research Triangle Park
Research Triangle Park, NC 27711

Number:
**G0436
Date:

Client Rep: Nazy Abousasedi
Work Order: G0436
PO Number: EP-W-05-030
Date Received: 04/04/2008

Project No:
Project Name: SOM Region II Modified Analyses
Case No: 37320
SDG No: B4KJ2

Test	Remarks	Matrix	Qty	Price	Test Total
SOM1.2_VOA_SIM_W	1543.0	Aqueous	17	$1.00	$17.00
SOM1.2_VOA_TRACE_W		Aqueous	17	$85.00	$1,445.00

Subtotal:	$1,462.00
Discount:	0.00%
Surcharge:	0.00%
Misc Charges:	$0.00
Payment Received:	$0.00
INVOICE Total:	$1,462.00

All invoices are due and payable net 30 days from receipt.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

SOP No. 30.0003

Rev. 12

	Signature	Date
QA Director:		10/17/08
Lab Director:		10/21/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

Rev. 12

1. Scope and Application

This Standard Operating Procedure describes the procedures that must be followed upon receipt of samples at MITKEM. It describes the procedures to log-in and store samples. It also describes procedures for checking samples out of the storage area for analysis, and for final disposal of samples. Detailed procedures for entering work orders and samples into Mitkem's Laboratory Information Management System (LIMS) are described in a separate SOP Number 20.0003.

2. Personnel Qualifications and Responsibilities

The Sample Custodian should have a two-year degree in environmental science or a related field or have six months of on-the-job experience working with a trained and qualified Sample Custodian.

3. Summary of Procedure

When sample coolers are received, cooler custody seals and temperature are checked. The chain-of-custody forms are signed, and the condition of the samples are checked, including any sample custody seals. Each sample is assigned a specific individual sample number. Any discrepancies are documented and resolved. Samples are logged-in to the Omega LIMS following procedures in SOP 20.0003. Labels are generated, and affixed to samples. The pH of aqueous samples (Inorganic only, never VOA) are checked and if need be, either acid or base is added. The addition of preservative(and its lot number) is recorded on the Condition Form. Samples are stored in secure area under documented custody conditions. Certain types of VOC soil samples require special handling, as described in **Section 8.2.2**. Samples are kept at Mitkem for a specific time as called for by contract with the client, and then disposed of in accordance with federal, state and local regulations. The door to the sample receiving room is locked at all times, using a keypad code lock.

4. Sample Preservation, Containers, Handling, and Storage

Samples received at Mitkem are collected by our clients. Mitkem does provide preservatives and sample containers pre-cleaned by bottle suppliers.

Tables 7-1, 7-2 and 7-3 of Mitkem's Quality Assurance Plan (QAP) lists the type of container needed for a particular type of analyte and the proper preservative.

5. Interferences and Potential Problems

Any problem with sample condition including custody seals, breakage, discrepancies in chain of custody or other shipping documentation, cooler temperature, missing samples, etc. must be communicated to the client promptly and resolved. The Sample Custodian or assistant documents the problem and notifies the Mitkem Project Manager, who will then notify the client. For CLP the Project Manager will notify the Sample Management Office (SMO).

6. Equipment and Apparatus

6.1 NIST-calibrated temperature "gun".

6.2 Condition Forms: Non-CLP, CLP (DC-1).

6.3 Orange colored Sample Condition Notification forms.

6.4 Narrow range pH paper, capable of recording pH 0- 2.5, and 11-13.

6.5 Computer with Accessories to run the LIMS Omega system.

6.6 Label printer and labels.

7. Reagents

7.1 Hydrochloric Acid (HCl), Fisher Trace Metal Grade.

7.2 Nitric Acid (HNO_3), Fisher Trace Metal Grade.

7.3 Sodium Hydroxide (NaOH), Certified A.C.S.

7.4 Zinc Acetate $(CH_3CO_2)_2$ Zn $2H_2O$, certified.

7.5 Sulfuric Acid (H_2SO_4), Fisher Trace Metal Grade.

7.6 Phosphoric Acid (H_3PO_4), Fisher Trace Metal Grade.

Reagents are ordered by the inorganic laboratory section, and kept in the sample receiving and bottle prep areas. Zinc Acetate is stored in the volatile laboratory as it is only used with volatile sample bottle prep.

If the reagent needed is in neat form (NaOH or Zinc Acetate) a new bottle may be obtained from the chemical storage area. If you take the next to the last or the last bottle for login dept use, you must inform the inorganic lab supervisor so more can be ordered.

8. Procedure

8.1 Log-In Procedures:

Samples received at Mitkem are received from private courier (FedEx, UPS, etc.) or Mitkem courier or delivery by the clients. All samples received must be accompanied by a chain-of-custody (COC) record.

8.1.1 Prior to opening coolers the receiving hood must be turned on, and safety glasses, lab coat, and gloves worn.

8.1.2 Select the next sequential red workorder folder with a pre-assigned work order number. Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. For example, 2006 is assigned the alpha character "E" and the 5th set of samples received during 2006 is assigned work order number E0005. Occasionally, a client wishes for a workorder number to be ongoing for several shipments of samples (i.e. EPA). The project manager will notify the sample custodian if additional samples are to be added to an existing workorder (an open Sample Delivery Group or SDG).

8.1.3 Before opening coolers, check the outside of the opening of the shipping cooler for the presence of custody seals. Remove any shipping air bills and note the work order number on them. This information should be noted on the Sample Condition Form (See **Attachments 1 and 2**).

NOTE: There are two different types of Sample Condition Forms; one for commercial samples, and one for the US EPA contract samples (see Attachments). Place air bills and all other paperwork for this shipment in the appropriate red workorder folder.

8.1.4 Open the cooler underneath the hood and remove the chain-of-custody form. This is typically located in a plastic zip-lock bag on the top of the samples or taped to the inside of the cooler lid. Write the Mitkem workorder number on

the COC. Sign the COC, noting the date and time of receipt of the samples at Mitkem. Look for return air bills to return coolers to the client, these are typically included in EPA projects. If the COC is missing, notify the Mitkem project manager using the Mitkem Sample Condition Notification (printed on orange paper) form immediately. Do not process the samples. Store the samples in the walk-in cooler until the client is contacted and responds.

8.1.5 Take the temperature of the contents using the temperature gun or thermometer.

8.1.5.1 Use the Temperature Gun as follows:

8.1.5.1.1 Unwrap a sample in the opened cooler. Unwrap enough of the sample in order to take a temperature reading.

8.1.5.1.2 Take the temperature by holding the gun no more than two inches from the sample container. Do not aim at the label or cap of the sample. Take several readings on at least 2 samples at opposite ends of the cooler. . Record the temperature on the lower right hand corner of the chain-of-custody or where noted on the Chain-of-Custody. The acceptable temperature range is 4° ± 2°C (2° to 6°C). If the temperature is acceptable, move on to **Section 8.1.6**. If the temperature is not within this range, enter the temperature on the Sample Condition Form, circle "yes" to the question regarding the Sample Notification form, and fill out the Sample Notification form (See **Attachment 3**). For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action guidance. Give the entire folder and the Sample Notification form to the project manager. The project manager will contact the client for instructions on how to proceed and return the folder to the sample custodian indicating action to be taken. During the interim, the samples in question will not be processed, but the closed cooler will be stored in the walk-in cooler.

8.1.5.2 If a temperature blank is present, use a thermometer as follows:

8.1.5.2.1 Insert the thermometer in the temperature blank.

8.1.5.2.2 Let stand for up to five minutes.

8.1.5.2.3 Read the temperature and record in the lower right hand corner of the COC or where noted on the Chain-of-Custody. If the temperature is not within 4° ± 2°C (2° to 6°C), proceed as in **section 8.1.4** above. For CLP projects, if the temperature

exceeds 10°C, the project manager will contact SMO for corrective action.

8.1.5.3 A calibration check of the thermometer is to be done quarterly or as the thermometer is replaced by comparing it's reading to the NIST thermometer and recording the results in the QA/QC Thermometer Calibration Logbook. See **SOP No. 110.0006** for the calibration procedure.

8.1.5.4 The presence/absence of a temperature blank in the shipment is documented on the Form DC-1. Follow the instructions on the DC-1 Form for contacting SMO, and for documentation in the event a temperature blank is not present.

8.1.6 If the Sample Custodian is not present, the person receiving the samples should perform all steps through **Section 8.1.5.2.5**, then store the cooler in the walk-in refrigerator, R1, until the Sample Custodian returns, after removing any VOC samples. Samples for VOC analysis require special handling, as described in **Section 8.2.2**. In general, if not logged-in immediately, VOA vials must be removed from the cooler and stored in refrigerator R2 until logged-in to the LIMS. If VOC samples are received in Encore devices (typically contained in silver foil pouches), or are noted as requiring freezing to preserve, they must be removed and stored in the freezer F2. Encore samples must be extruded into pre-weighed VOC vials as soon as possible. Contact one of the following to insure this occurs: VOA Laboratory Supervisor, Sample Custodian, Project Manager, Laboratory Operations Manager or QA Director. Leave the paperwork in the red folder on the sample custodian's desk.

8.1.7 Once all of the coolers received have been inspected as above, prioritize them for logging into the LIMS with fastest turn-around times and short holding times processed first. The labs should be notified immediately about quick-turn analyses. This can be done either verbally, or by copying the COC and distributing that to the laboratories.

8.1.8 Remove and inspect samples from the coolers for the first workorder. Sample containers are removed from the cooler and lined-up on the counter in the same order as listed on the chain of custody form. Check to see that the correct number of samples is present. Check to see if any samples are broken. Check aqueous VOA sample vials for the presence of bubbles larger than the size of a pea. If bubbles are larger than the size of a pea, a Sample Notification form should be filled out and given to the Project Manager. Check to see that the quantity present is sufficient for the test requested. Make sure the client IDs on the bottles agree with the COC. Check the custody seals, if any, on the sample containers to ensure the all seals are present and intact.

8.1.9 For U.S. EPA samples, sign the COC and note the temperature on the bottom right corner of the COC. Place the project number in the top right hand corner of the COC. Record the following information on the Form DC-1.

- Sign DC-1. Note that the person signing should be the person who signed for receipt on the COC. There will be one DC-1 form for each cooler received for a particular case. Only the samples in that particular cooler will be listed on that DC-1 form.
- EPA Case number from the COC.
- List sample tag control numbers and compare these with the COC records.
- Document whether or not these numbers agree.
- If Sample tag numbers are not listed on the COC form, record this fact.
- Record the custody seal numbers if present.
- Write the work order number on the air bill and place it in the red project folder. Record the name of the courier and the air bill number on Form DC-1.
- Date of receipt.
- Time of receipt.
- EPA sample numbers.
- SDG-Final Sample must be documented on EPA TR/COC.
- SDG (Sample Delivery Group) number. The SDG is the lowest alphanumeric sample on the first shipment and remains the same until the work order is closed.
- EPA sample numbers.
- Assigned Lab ID numbers. Note that with EPA projects, shipments may be received over several days to be added to the same work order, up to twenty field samples, not including PE samples. The SDG number stays the same, but the sample ID numbers run consecutively.
- Whether samples were delivered by hand or not.
- Any problems and discrepancies in "remarks" box.
- The cooler temperature is entered in item #10 of the Form DC-1.

Note from the COC whether the work order is closed, or if more samples are expected to be added (work order/SDG is still open). EPA projects are closed automatically after one week from the date of receipt, for cases with a turnaround time of 14 or 21 days and three days for samples with a turnaround time of 7 days, of the first shipment or sooner if the client checks the box that the project is closed.

Discrepancies in EPA shipments should be reported to the Project Manager directly. Any irregularities in the above information should be documented and the client contacted by either Project Manager or the Sample Custodian. Until an answer is received from the client, samples should be stored in the walk-in cooler.

If there are any discrepancies, note them on the Sample Condition Notification Form and give the form and the workorder folder to the project manager. Samples with

issues will not be processed further, but stored in the walk-in cooler until a resolution has been reached with the client. For CLP (EPA) samples, the Sample Management Office (SMO) will be contacted for corrective action. Communication of the corrective action will be placed in the red project folder.

8.1.10 Log the workorder and samples into the Omega Laboratory Information Management System (LIMS) following procedures described in SOP 20.0003. Labels are generated and attached to each sample container. In the case of pre-preserved samples in VOA vials (sodium bisulfate, DI water and/or methanol), be sure the Mitkem sample ID label does not cover the tare weight of the vials.

8.1.11 Have the project manager or a second person check the Lab IDs on the labels vs. the client IDs on the COC to confirm the bottles have been properly labeled. Ensure the reviewer initials the proper line of the Sample Condition Form or DC-1 form.

8.1.12 Enter the workorder number, date received, and client name in the Sample Receipt and Tracking Logbook (**Attachment 6**). Occasionally, a client wishes for a project number to be ongoing for several shipments of samples (i.e. EPA). In such cases the date of arrival of each subsequent shipment should be noted for the original work order number.

8.1.13 Inorganic preserved samples must now be tested for pH as follows:

For metals, wet chem, etc., pour a small amount of sample over a strip of pH paper that is suspended over the mouth of a waste container. The pH of the acidified samples are taken by using the narrow range pH strips which measures pH 0 to 2.5. The sample pH should be less than two, as indicated by the color on the pH indicator package. The pH of the basic samples are taken by using the narrow range pH strips which measures pH 11.0 – 13.0. The basic samples should have a pH 12 or greater, as indicated by the color of the pH indicator package. (See **Attachment 4** for instructions on the use of the pH paper.) Acidified samples must have a pH of less than 2. Any pH adjustments that need to be made are recorded on the Sample Condition Form for the workorder. Record the lot number and ID of the reagent used and the approximate volume used to adjust the pH. For metals samples requiring acidification, alert the inorganic laboratory to allow 24 hours before metals preparation to allow enough time for the acids to dissolve any metals that adsorb to container walls.

8.1.14 Some samples (i.e. fecal coliform bacteria) which require subcontracting must be done so immediately as the analysis has an extremely short hold time. A list of analyses typically subcontracted can be printed from the LIMS Omega system. An Outside Services Purchase Order is also generated from the LIMS system when the batch of samples to subcontract is created.

8.1.15 Receiving personnel make one copy of the sub contract COC. This COC accompanies the samples and is signed by the laboratory that will perform the analysis. A copy of the COC is placed into the red workorder folder and one copy is sent to Accounting.

8.2 Storage and Location of Samples:

8.2.1 Semivolatile Organic, Pesticides/PCBs, and Wet Chemistry Samples:

Samples are stored in the main walk-in refrigerator (R1) in the sample receipt room at 4° ± 2°C. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook . The person putting the samples into the walk-in must put their initials in the logbook beside the location entries.

8.2.2 Volatile Organic Samples:

Samples for VOA are stored separately from all other samples, in refrigerators or freezers dedicated for this purpose. These refrigerators and freezers are located in the VOA Laboratory. A soil sample for VOA may contain one or more of the sample types listed below. Care must be taken to insure each sample vial type receives the proper handling. Soil samples for volatiles analysis must have special handling performed by the VOA lab staff.

A storage blank must accompany all VOA samples requiring EPA/CLP or New York State ASP analyses when placed in the VOA refrigerators. The storage blanks are prepared in the volatiles department using the VOA Type I water. The 40ml vial is filled so there is **no** headspace, capped immediately and tightly, and labeled with the date prepared and any preservative added. The storage blanks for unpreserved low/medium soil samples are prepared by placing approximately 5 g of inert sand in dry closed-system purge-and-trap vials. These inert sand storage blanks will be stored in the same freezer with unpreserved low/medium soil samples.

8.2.2.1 Samples suspected of containing high concentrations of volatile organics must be stored in a separate VOA refrigerator designated for such samples. This isolates these high concentration samples from other samples, and helps to control cross-contamination.

8.2.2.2 Soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial) should be stored in a VOA freezer (containing no standards or sample extracts). These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.3 Soil samples that contain up to 5 mL of water. These vials are also to be stored in a VOA freezer. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.4 Soil samples that contain 5mL of water containing a sodium bisulfate preservative. These vials are also to be held in a VOA freezer (specifically for EPA CLP samples). Samples for Method 8260 analysis may be held in a VOA refrigerator. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.5 Soil samples received in the Encores (typically contained in silver foil pouches), the samples must be extruded into tared, dry closed-system purge-and-trap vials by the VOA lab staff, then re-weighed to obtain the final sample weight. This should happen immediately (for EPA CLP samples), but no longer than 48hours of sample collection. The vials are then to be placed into a VOA freezer.

8.2.2.6 Soil samples may also arrive as methanol preserved samples. These samples do not require freezing, but they shall be held in a VOA refrigerator to be weighed by VOA lab staff prior to analysis. If possible, check that the methanol covers the soil samples within the vial. If the methanol is insufficient, note this on the Sample Condition Form and relay the information to the VOA lab.

8.2.2.7 Unpreserved VOA soil samples should be stored in a VOA freezer (EPA CLP samples) or a VOA refrigerator (for Method 8260 samples) until the time of analysis.

8.2.2.8 Water samples are stored in VOA refrigerators. Trace Volatile samples for the EPA/CLP SOM01.2 contract must be stored in a separate refrigerator from all other samples. At present, this has been designated as **R13 and R4**.

8.2.2.9 Log the samples into the VOA lab. Enter the workorder and other pertinent information in the Volatile Receiving Logbook (See **Attachment** 7) and sign off that the samples have been relinquished. If the samples are ever removed from VOA they must be signed out of the VOA lab as well.

8.2.3 Metals Samples:

Aqueous preserved samples for metals analyses are stored on the rack in the sample receipt area. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook . The

person putting the samples onto the rack must put their initials in the logbook beside the location entries.

Standards must be stored in separate freezers or refrigerators isolated from samples and sample extracts.

8.3 Storage and Location of Extracts:

Extracts for semivolatile analyses must be stored, shielded from light, at $4^{o} \pm 2^{o}C$ in a separate refrigerator stored either in the laboratory or in Unit 3. Extracts for Pest/PCB are stored in refrigerators in the same manner.

Extracts must be stored separately from standards.

Extracts for semivolatiles and Pest/PCB must be stored until the proper time to dispose of them per client approval. Mitkem normally retains sample extracts for a period of three months from the date of delivery of the final report to the client

US EPA CLP and New York State ASP extracts must be stored for 365 days after delivery of a complete data package to the client.

8.4 Temperature Monitoring:

The temperature of the storage areas must be monitored on a daily basis, including weekends and holidays. This temperature must be entered in the Temperature Logbook. If any refrigerator fails, first investigate the problem. Was the door ajar? Is something preventing the door from shutting fully? If the situation can be rectified, do so and record the temperature again after a sufficient period of time. If the deviation occurred at the end of the day, re-record the temp as soon as possible the following morning. When the temperature meets criteria, document the issue and note that the refrigerator/freezer has been "*returned to control*". If there is no obvious issue with the refrigerator/freezer, or the refrigerator does not maintain the correct temperature, the QA Director or Lab Director must be notified immediately to correct the problem. Fill out a report in the LIMS Omega Corrective Action Report option. Acceptable temperature ranges are $2^{o} - 6^{o}C$ for refrigerators and $-10^{o}C$ to $-20^{o}C$ for freezers. The Sample Receiving supervisor or his/her designee will check and record the storage area temperatures daily.

8.5 Tracking of Samples and Extracts:

8.5.1 Samples:

8.5.1.1 Sample Log-out:

When samples are taken from their storage areas for extraction, they must be signed out using the appropriate Sample Receipt Logbook. The log-out procedure is a two-person procedure that requires samples to be relinquished by either the Sample Custodian or Authorized Laboratory Personnel

The analyst retrieves the samples from the walk-in cooler and the Custodian or Authorized Laboratory Personnel checks the sample numbers. The Mitkem sample numbers as well as the bottle # are recorded (i.e. E0140-02B#2). Both people initial and date the Receiving Logbook in the appropriate places. When the sample is logged-out by an analyst, it is considered to be in the custody of that analyst until he/she returns the sample to its storage area.

Upon the sample's return to the storage area the samples are signed into Receiving using the same procedure. This time the analyst relinquishes the samples and the Custodian or Authorized Laboratory Personnel checks the sample numbers to be returned. Each person signs and dates in the appropriate places in the Receiving Logbook.

Authorized laboratory personnel are employees who are knowledgeable in regard to the receipt and log-in of samples. A list of Authorized Laboratory Personnel is posted on the Receiving bulletin board. (See **Attachment 5**).

8.5.1.2 Sample Extraction:

If a sample is signed out for extraction, the sample is logged into the lab by entering the Lab ID into the appropriate extraction logbook. After extraction, any remaining sample volume is returned to the storage area and signed back into the Receiving area for storage as noted in **Section 8.5.1.1**.

When working with samples in the preparation laboratories, the following procedures must be adhered to:

- All activities performed on the sample must be recorded.
- Entries in the logbooks must be signed and dated (mm/dd/yy).
- All entries must be made in ink.
- Corrections and additions must be made by drawing a single line through the errors, entering the correct information, and initializing and dating the new information.
- Unused portions of the logbooks must be lined-out or "Z-ed" out.
- Logbook entries must be made in chronological order.
- For US EPA/CLP samples, entries are recorded for only one SDG on a page, except in the event where SDGs share quality control samples.
- Information inserted in the logbooks must be permanently affixed, signed, and dated across the insert.
- Copies of all pertinent logbook pages must be placed in the workorder file.

° Proper subsampling techniques can be located in the Subsampling SOP, 110.0039.

8.5.1.3 Extracts/Digestates:

The organic sample extracts, once completed by the preparation laboratory, are transferred to the appropriate instrumentation lab and signed into the extract transfer logbook. The analyst will sign for the extracts and enter storage location information. Inorganic metals and mercury digestates are transferred to the instrumentation lab. The analyst signs off on the preparation logbook at time of transfer.

When the time period for organic extract storage has expired, extracts may be disposed of in the same manner as would used/expired standards. CLP extracts must be stored for one year. The transfer and disposal for these extracts must be documented in the Extract Disposal Logbook.

When the time period for inorganic digestate storage has expired, digestates are disposed in the same manner as would used/expired inorganic standards.

See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the actual disposal of above extracts/digestates.

8.5.1.4 Sample Disposal:

The unused portion of sample is returned to the Receiving area for long term storage. Such portions must be stored for a certain length of time as required by client contract and then properly disposed. Unused sample is disposed with the use a licensed hazardous waste contractor, currently Univar. Routinely, one of the sample custodians runs the LIMS Omega Disposal program to produce a list of samples ready for disposal. See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the different wastestreams and disposal options.

All soil samples received from outside of the 48 contiguous United States must be disposed of following special procedures outlined by the USDA, as detailed in Mitkem SOP No. 30.0024 Sample and Waste Disposal section 8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

10.1 The Sample Custodian will calibrate the temperature gun using an NIST calibrated thermometer on a quarterly basis. The correction factor will be noted on the temperature gun and recorded in the NIST Thermometer Calibration Logbook. The serial number of the NIST thermometer will also be recorded in the logbook.

10.2 On a daily basis, the Project Managers will review that the Sample Condition Forms are filled out correctly.

10.3 Twice daily (typically early morning and mid-day) the temperatures of the refrigerators and freezers will be checked and the temperatures recorded. Deviations will be noted in the logbook. If after 36 hours the temperature problem cannot be corrected with manual adjustments, the refrigerator or freezer will be replaced.

10.4 The Hazardous Waste Inspection Logbook will be completed weekly, after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11. Data Validation and Reporting

A copy of the chain of custody and Sample Condition Notification form accompanies the data reports to the client. Also any communication records are to be included in the data package (telephone conversations, e-mail communication, etc.).

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range. Notification reports (orange sheets) are generated in the event of an out-of-control situation occurs at sample log-in that cannot be corrected. Project Managers contact the client in order to ensure what type, if any, corrective action needs to be taken. Other corrective actions concerning the Receiving Area are recorded using the Omega LIMS Corrective Action Report.

13. Health and Safety

The sample custodian must wear a lab coat, safety glasses, and gloves when handling samples and preservative chemicals. Samples coolers should be opened under a hood. All samples should be handled as though they are potentially hazardous. The outside of sample containers should also be handled as though they are contaminated. Any broken sample containers must be handled very carefully to prevent exposure to the sample.

14. Pollution Prevention, Waste Management, and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

US EPA CLP SOW SOM01.2
US EPA CLP SOW ILM04.3
US EPA CLP SOW ILM05.4
US ACOE Shell for Chemical Analytical Requirements, Appendix H

Attachments:

1. **Attachment 1**: Mitkem Sample Condition Form.
2. **Attachment 2**: EPA Sample Condition Form (DC-1).
3. **Attachment 3**: Sample Condition Notification Form.
4. **Attachment 4**: Instructions for the use of pH paper.
5. **Attachment 5**: Example Authorized Personnel list.
6. **Attachment 6**: Sample Receipt and Tracking Logbook.
7. **Attachment 7**: VOA Receiving Logbook

Attachment 1
Mitkem Sample Condition Form

MITKEM LABORATORIES
Sample Condition Form

Received By:	Reviewed By:	Date:	MITKEM Workorder #:
Client Project:		Client:	

		Lab Sample ID	Preservation (pH) HNO_3	H_2SO_4	HCl	NaOH	H_3PO_4	VOA Matrix	Soil Headspace or Air Bubbles ≥ 1/4"
1) Cooler Sealed	Yes / No								
2) Custody Seal(s)	Present / Absent								
	Coolers / Bottles								
	Intact / Broken								
3) Custody Seal Number(s)									
4) Chain-of-Custody	Present / Absent								
5) Cooler Temperature									
Coolant Condition									
6) Airbill(s)	Present / Absent								
Airbill Number(s)									
7) Sample Bottles	Intact/Broken/Leaking								
8) Date Received									
9) Time Received									

Preservative Name/Lot No:

VOA Matrix Key:

US = Unpreserved Soil	**A** = Air
UA = Unpreserved Aqu.	**H** = HCl
M= MeOH	**E** = Encore
N = $NaHSO_4$	**F** = Freeze

See Sample Condition Notification/Corrective Action Form yes / no

Rad OK yes/ no

Attachment 2

EPA Sample Condition Form (DC-1)

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name				Page 21 of 32
Received By (Print Name)				Log-in Date
Received By (Signature)				
Case Number		Sample Delivery Group No.		Mod. Ref. No.

Remarks:			Corresponding		
		EPA Sample #	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____ _____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

SAMPLE LOG-IN SHEET

Lab Name	Page 22 of 32
Received By (Print Name)	Log-in Date
Received By (Signature)	

Case Number	Sample Delivery Group No.	SAS Number

Remarks:		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Chain of Custody Records	Present/Absent*				
4. Traffic Reports or Packing Lists	Present/Absent*				
5. Airbill	Airbill/Sticker Present/Absent*				
6. Airbill No.	_____ _____				
7. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
8. Sample Condition	Intact/Broken*/ Leaking				
9. Cooler Temperature	_____				
10. Does information on custody records, traffic reports, and sample tags agree?	Yes/No*				
11. Date Received at Lab	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

Attachment 3
Sample Condition Notification Form

Sample Condition Notification

Mitkem Project#:_____ **Date of Receipt:**_____
Client:_____ **Received By:**_____
Client project #/name:_____

Unusual Occurance Description:

_____

_____

_____

_____

_____

_____

Client Contacted:

Contacted via: Phone/Fax/E-mail
Date:_____ **Time:**_____
Contacted By:_____
Name of person contacted:_____

Client Response:

Responded via: Phone/Fax/E-mail
Date:_____
Name of person responding:_____
Responding to:_____

_____

_____

_____

_____

_____

Mitkem Action Taken: _____

_____

_____

_____

_____

Attachment 4

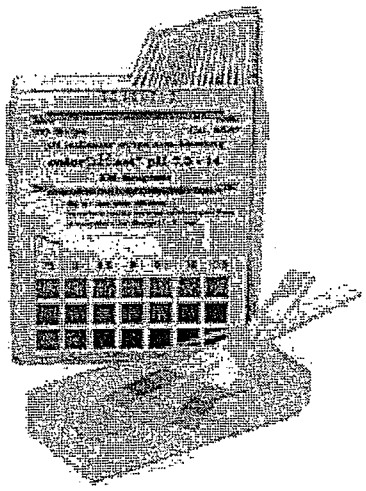
Instructions for use of pH paper

General Procedure for the Use of colorpHast® pH Test Strips

1. Remove a strip from the box.
2. Immerse the reaction zone of the strip into the solution for approximately 30 seconds to 1 minute, allowing enough time for the reaction to take place.
3. Remove the strip from the solution, wiping the strip along the edge of the vessel to remove excess liquid from the strip.
4. Compare the strip's reaction zones to the color chart on the box while holding the strip to the top of the box and the reaction zones to the bottom of the box.
5. Record the results of the closest matching color to the strip.

- *For additional application or product information, ordering information or other technical questions, please call our Technical Service Department at:*

  1-800-222-0342 *or* **1-856-423-6300**



EMD Chemicals Inc.
480 South Democrat Road
Gibbstown, NJ 08027

Phone: 856-423-6300
Fax: 856-423-4389
www.emdchemicals.com

Attachment 5

Example Authorized Personnel list

Sample log-in/out: **Authorized Personnel**		
Name	**Dept**	**Initials**
Anderson, Courtney	Inorganics	*CJA*
Badura, Karolina	PEST/PCB	*KB*
Chiu, Kin	Mitkem	*KC*
Cardoso, Antonio	Oprep	*AC*
Ding, Yihai	Mitkem	*YD*
Ding, Xiangdong	VOA	*XD*
Gaudreau, Veronica	Receiving	*VEG*
Jaffer, Nathan	Inorganics	*NJ*
Healey, Josh	Receiving	*JH*
Kaczorowski, Przemyslaw	Oprep	*PK*
Lawler, Andrew	Oprep	*AEL*
Lawler, Edward	Project Management	*EL*
Lawler, Sharyn	QA/QC	*SBL*
Leveillee, Roy	Oprep	*ROY*
Luo, Wei	VOA	*WL*
Lucas, Derek	PEST/PCB	*DL*
Maczewska, Beata	SVOA	*BM*
Marquis, Ashley	VOA	*ALM*
McGorty, Cassandra	Inorganics	*CJM*
McGovern, Kerri	Inorganics	*KLM*
Mosher, Cassie	SVOA	*CLM*
Montmarquet, Timothy	Mitkem	*TM*
Nadeau, Cory	Receiving	*CAN*
Ng, Agnes	Project Management	*ARN*
Ng, Shirley	Project Management	*SN*
Rosadzinski, Tomasz	Oprep	*TR*
Sawyer, Tom	Inorganics	*TS*
Smart, Dawne	Inorganics	*DES*
Wetherbee, John	Inorganics	*JRW*
Wortley, Stephen	SVOA	*SW*
Veilleux, Brian	Inorganics/Oprep	*BV*
Wieczorek, Agnieszka	PEST/PCB	*AW*
Zharkova, Sofya	PEST/PCB	*SZ*
Zhao-Anderson, Heather	VOA	*HZA*

Most common personnel using log-in/out

Attachment 6

Sample Receiving and Tracking Logbook

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc.

Sample Receiving Logbook

Workorder No. _____

Client Name: _____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

OUT		IN	
Relinquished By	**Received By**	**Relinquished By**	**Received By**
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			

Comments: _____

_____

Please record analyst's initials, date, and sample #s removed. Add any comments if necessary (broken bottles, empty jars, etc.)
Include the abbreviated name of the test to be performed., ie: SVOA, PCB...near the "samp. #s".
Include bottle or jar number when more than one.

Reviewed:_____

Logbook ID: 30.0287-09/08

Attachment 7

Volatiles Receiving Logbook

MITKEM LABORATORIES: VOLATILE SAMPLES RECEIVING LOGBOOK

VOA Log-In Date	Workorder	Client ID	Sample Numbers	Relinquished by:	Received by:	Pres. Used	F/R	Returned to R1

Logbook ID 90.0191-06/08

Reviewed By:

"Preservative Used" Key

UA = Unpreserved Aqueous	**H** = HCL	**A** = Air	**M** = MeOH	**E** = *Encore
US = Unpreserved Soil	**N** = $NaHSO_4$		**F** = Freeze	**T** = Trace, HCL

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP No. 30.0024

Rev. 8

	Signature	Date
QA Director:		10/17/08
Lab Director:		10/21/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP 30.0024 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the procedures for the disposal of the unused portion of samples submitted for analysis, as well as solid and aqueous wastes and sample extracts to minimize the amount of waste in the lab and organize the manner in which it is handled.

2. Personnel Qualifications and Responsibilities

Personnel handling samples must be aware of the potential dangers posed by the samples, and handle them properly to minimize this danger. Personnel handling samples must also maintain the documented chain of custody of the samples from the time they are received at Mitkem until they become laboratory waste. Laboratory waste must be handled in a safe manner to minimize any exposure to this material.

3. Summary of Procedure

Waste stream manifest profiles have been identified by Mitkem and the waste-disposal company. These profiles dictate how to dispose of each individual waste, as described in this SOP. If any question arises regarding the disposal or classification of waste, the Waste Disposal Technician, or senior laboratory management should be consulted.

Once sample analyses are complete and a final report has been issued to the customer, samples are stored for a specified length of time before disposal. This timeframe is usually dictated by the client. Strict chain of custody documentation is maintained for these samples until samples are disposed into one of several waste streams. This waste is removed from Mitkem by a licensed waste-hauler for final disposal.

Safety equipment will be worn prior to and during any waste disposal. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Waste drums. Polyethylene drums are used for liquids (acids or basic/spent CN solutions) and steel open top drums are used for waste solid materials.

Burp-free Ultra Funnel, various sizes, polyethylene funnel used to allow safe pouring of liquid wastes into drums. Product is vented and has locking lid.

Spill kits

Drum Pump

Drum Lift

Satellite Nalgene storage containers with lidded funnels for interim liquid waste storage in Wet Chem, Volatiles and Metals Labs.

Satellite storage containers (red metal Justrite Oily waste Can ,10G capacity) for interim solids storage in Organic Prep, Semivolatiles and Volatiles Labs.

Satellite storage container (Justrite Safety Disposal Can, 2G capacity) for solvent waste in Volatile Lab.

7. Reagents

Not applicable.

8. Procedure

8.1 Classification of Waste:

Laboratory waste products fall under one of the following categories:

8.1.1 Methylene Chloride, Methanol, Water:

Waste solvents are emptied into a solvent drum located in the organic sample preparation laboratory. This waste consists primarily of methylene

chloride with lesser portions of methanol and water. Trace amounts of other solvents may also be included from time to time. Due to the high proportion of methylene chloride in the waste, the material is flammable, and is classified as D001-type waste. Whenever a new drum is started, a Hazardous Waste label is attached to the outside.

When the drums are full the waste drum is taken to the Hazardous Waste storage shed. An accumulation date is added to the Hazardous Waste label on the drum.

Empty solvent bottles are evaporated to dryness and discarded into the general solid waste dumpster (garbage). Glass containers that present a risk of injury should be discarded into a broken glass container for disposal.

Any lab apparatus, gloves, paper towels, etc. exposed to methylene chloride must be discarded into the red solid waste drums in the Organic Prep Lab. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.2 Acids, Water:

Acid waste consists of approximately 98% water and 2% acid. This waste stream also includes the remainders of spent or expired metals standards, digested samples, TCLP and SPLP leachates and undigested aqueous samples preserved with acid. This waste is emptied into polyethylene acid waste drums in the hazardous waste area. Acidic waste from the Lachat analysis of nitrate/nitrite or chlorides are stored temporarily in Unit 3 in a small waste container equipped with a closing safety funnel. The container is routinely emptied into this same wastestream. A Hazardous Waste label is affixed to the side of the drum and an accumulation date is recorded on it the first time the drum is started. This waste is classified as D002 type waste. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.3 Scintillation Vials, Methylene Chloride, Hexane:

This waste stream consists of Organic analysis vials used for GC and GC/MS analyses. Spent or expired organic standards in solvent are also included in this wastestream. The vials are disposed of unopened. Vials are initially disposed in the GC Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone:

This waste stream contains the remainders of solid and soil samples. This waste stream also includes laboratory solid waste that has been stored in the red metal drums in the Organic Prep Lab. When full the laboratory red metal containers are taken to the Hazardous Waste shed and the contents dumped into the waste solids drum. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and is later removed by the Waste Hauler within 90 days of the accumulation date. This waste has no RCRA Characterization code.

Any solid matrix samples received from outside of the 48 contiguous United States, or from any other area identified by the client as a US Department of Agriculture Soil Quarantine Area must have special disposal in order to kill any quarantined organism. Samples identified as such, along with their entire container including jar cover, are placed in a disposable aluminum pan and placed in the kiln (in Unit 3) and heated to 400 degrees for 30 minutes. After the pan has cooled, the sample(s) and aluminum pan(s) are disposed in the waste soil/solid drum.See **Attachment 1** for incineration procedures.

8.1.5 Glass Vials, Methanol, Water, Soil:

This waste stream consists of primarily 40-ml organic analysis vials used for VOA analyses. The vials are disposed of unopened. Vials are initially disposed in the VOA Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.6 Spent Carbon with Methylene Chloride:

This waste stream contains the remainders of Mitkem's discharge. Discharge is run through tanks of carbon material as a means of removing methylene chloride. When the tanks are fully deactivated, the carbon is removed and dumped into a steel drum. The tanks are then filled with activated carbon. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and the drum is later removed by the Waste Hauler within 90 days of the accumulation date. This waste stream is not associated with any RCRA Characterization code.

8.1.7 Electronic Device with Beryllium Oxide:

This waste stream contains the power tube of an ICP spectrophotometer. Because of beryllium in the tip of the tube, it cannot be disposed of with

ordinary trash. The tube is placed in a sealed cardboard box and a Hazardous Waste label with an accumulation date is affixed to the outside of the box. The box is later removed by the Waste Hauler. This waste stream is not associated with any RCRA Characterization code.

8.1.8 COD Vials:

This waste stream consists of glass tubes that contain a mixture of mercuric sulfate, silver sulfate, sulfuric acid, and chromic acid. The tubes are used for COD analyses and are disposed unopened in their entirety. Vials are initially disposed in the Wet Chemistry Laboratory in a covered cardboard box with a Hazardous Waste Label. When this box is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D002, D009, D011 waste.

8.1.9 Lab Pack Waste:

From time to time, waste or unused chemicals (non-solvents) will require disposal. These materials are segregated and placed in a lab pack container. An inventory of materials is maintained for the lab pack drum. The materials are stored in the laboratory in a closed cardboard box with a Hazardous Waste label attached. Consult the waste hauling company when sufficient volume of material is present in the laboratory to initiate a lab pack.

8.1.10 PCB Waste:

Any PCB-containing material (at a concentration of greater than 50 ppm) must be segregated and disposed according to PCB-specific procedures. This material would generally consist of any oil sample containing over 50 ug/g (ppm) of PCB. Samples containing this high concentration of total PCB will be identified by the GC Laboratory Manager or the Laboratory Director. The container for this sample will be prominently labeled "for return to client" when the holding period (as identified in **Section 8.2.1** below) is reached. The Mitkem project manager will be notified, as will the client, with a notation in the report Project Narrative or Cover Letter that the sample will be returned.

8.1.11 Water/Sodium Hydroxide/Pyridine:

This waste stream consists of sulfide and cyanide digestates and spent standards. Pyridine waste from the analysis of Cyanide (Lachat), and basic pH Ion Chromatography (IC1) wastes are stored temporarily in Unit 3 in several small waste containers equipped with closing safety funnels.

The containers are routinely emptied into a polyethylene Basic Waste drum in the hazardous waste area. This waste is classified as D002, D038 waste

8.2 Procedure for Disposal of Sample Remainders:

8.2.1 Determination of Disposal Date:

Samples are held for a period of time following receipt at the laboratory or release of the final report to the client. Mitkem's policy is to hold samples for a specified length of time. On a case-by-case basis, arrangements may be made between the client and Mitkem through discussion with the Mitkem Project Manager. This agreement is then saved in LIMS in the individual Client Project. Default values are used otherwise. To review the Sample Hold Times and Disposal Options on a particular workorder, go to the Report Option at the Workorder Main screen.

For CLP, samples are held for 60 days after delivery of the reconciled data package. Sample extracts must be stored at 4°C until 365 days after delivery of a complete, reconciled data package to EPA.

8.2.2. Documentation of Sample Disposal:

When the specified hold time has passed since the receipt date, the samples can be disposed. Sample receiving personnel select sample remainders from their storage locations and remove these to a designated location outside of the sample receiving area for disposal by Sample Receiving or laboratory personnel. Documentation that sample remainders have been disposed is maintained in the Mitkem Omega LIMS. When samples are removed from storage for disposal the person removing them enters the disposal date and their initials into LIMS.

8.2.3. Final Disposal and Record keeping:

All samples are to be disposed of according to **Section 8.1**.

Once waste drums are full, approximately bi-monthly, the waste hauling company is called to schedule a pick-up. Mitkem currently uses Univar to arrange for disposal of laboratory waste. Univar will remove the waste from the Mitkem storage area, and arrange with Pollution Control Industries (PCI) in Michigan for ultimate disposal of the waste material. Waste manifests are initiated by the hauler, signed by the QA Director or his designee when drums are loaded on the hauler's truck, signed and mailed back to Mitkem following final disposal. Manifests are filed

according to date in the QA office. Mitkem's Generator Number is **RI5000009621**.

9 Data Reduction and Calculations

Not applicable.

10 Quality Assurance/Quality Control

The Hazardous Waste Inspection Logbook will be completed weekly after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11 Data Validation and Reporting

After sample disposal, the disposal dates are entered into the LIMS for each workorder. In addition, the samples'storage location is changed to "disposed".

12 Corrective Action Procedures

Corrective actions concerning sample or waste disposal are recorded using the Omega LIMS Corrective Action Report under the Receiveing Department or another lab area involved in the issue. Corrective actions should implemented in the event of an out-of-control situation involving sample or waste disposal that cannot be easily corrected. The sample custodian should contact the Safety team, Hazardous Waste Coordinator or Lab Director for help determining what corrective action needs to be taken.

13 Health and Safety

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the Mitkem Safety Manual. Bottle or container labels also provide important information that must be noted. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

All laboratory employees who either sign a Hazardous Waste manifest or handle hazardous waste directly must attend an annual RCRA training class. At this time Summit Technologies of New Hampshire provides the necessary on-site training for Mitkem's laboratory personnel.

14 Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15 References

None.

Attachments:

1. **Attachment 1**: Instructions for the Kiln.

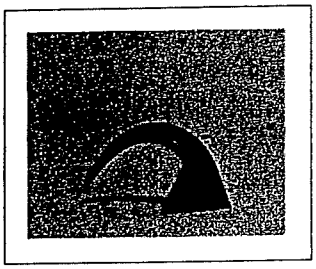
Attachment 1

Instructions for the Kiln

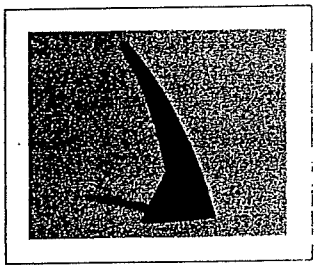
To use Cone-Fire Tuning, **IdLE** must display. Press **ENTER**, then **1. ConE** will appear. Press **999, ENTER. rSLt** (for result) will display alternating with the current Tuning number. Enter the new number, then press **ENTER. ConE** will appear. Continue entering the values for the Cone-Fire program in the usual way. Once you change the Tuning number, Cone-Fire will remain adjusted to that number until you change it again.

The large cone on the kiln shelf should be visible through a peephole. Avoid exposure to cool air by keeping the cone at least 3" away from the peephole. Program the DTC 800C for the cone on the shelf and fire. After cooling, check the cone:

The cone bent to 6 o'clock: In this case, the controller is matched to your kiln.

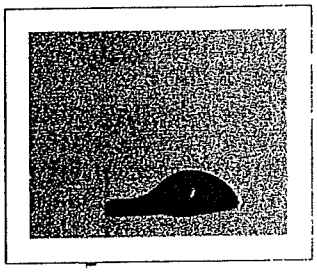


The cone did not bend far enough: Use a lower Tuning number for the next firing.



The cone bent too far: Use a higher Tuning number for the next firing.

Do not be overly concerned with achieving an exact 6 o'clock bend. The difference between a 3 o'clock and a 6 o'clock bend is only a few degrees. Cones, even from the same box, also vary slightly.



Ramp-Hold Mode

Ramp-Hold is available on both the DTC 800C and DTC 800.

In Ramp-Hold, the kiln fires in segments, or stages. Each segment has a firing rate, firing temperature, and hold (or soak) time. Ramp-Hold includes eight segments. Use only the number of segments you need per firing, from one to eight.

Rate is degrees of temperature change per hour, from 1° to 9999°F. (e.g., to increase temperature by 500° per hour, enter a rate of 500).

To control cooling, set the segment to a lower temperature than that of the preceding segment.

Storing User Programs in Ramp-Hold

A user program is a set of firing instructions to run the kiln in Ramp-Hold. A user program includes number of segments needed; firing rate, temperature, and hold (if any) for each segment; and an alarm temperature (if any).

Ramp-Hold Programming

As the program prompts you for segments, rate, temperature, etc., you will see values from the last firing. To use these again, just touch **ENTER**.

To fire without the Delay feature: Follow steps 1 through 9. Then press **START** twice.

1 Turn controller safety switch ON.

2 If controller displays **ErrP** or a flashing temperature, touch **ENTER. IdLE** will appear.

3 Touch **ENTER** then **4. USEr** will appear. Enter a number from 1 to 6 for the stored program desired. ENTER 1

4 Touch **ENTER. SEGS** will appear. Enter number of segments you will use. ENTER 1.

5 Touch **ENTER. rA 1** will appear. Enter firing rate for segment 1 (temperature change per hour: any temperature from 1° to 9999° F.). ENTER 400°C.

6 Touch **ENTER. °F 1** will appear. Enter the temperature you will be firing to in segment 1. ENTER 250°C

7 Touch **ENTER. HLd1** will appear. Enter segment 1 hold (soak) time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). ENTER 00.20

8 Touch **ENTER.** Continue entering values for all segments.

9 Touch **ENTER. AlAr** will appear. Enter alarm temperature. (Enter 9999 to turn alarm off.) Then touch **ENTER.**

IdLE will appear.

10 To set Delay Fire, touch **ENTER** then **3. dELA** will appear. Enter delay time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). Then touch **ENTER.** (Delay zeroes out after each completed firing.) NO DELAY

11 To start program, touch ENTER twice. **-On-** will appear, then kiln temperature. If a delay was programmed, **-On-** will appear, then time remaining until start.

To stop the program during the firing cycle, touch **STOP** or turn the safety switch OFF. When program fires to completion, **CPLt** will appear alternating with total firing time in hours and minutes. Turn safety switch OFF when **CPLt** appears. To shut off the alarm when it sounds during a firing, press **ENTER.** When Temp decreases after the 20 minutes @ 250°C, hit STOP. LET COOL DOWN ON ITS OWN.

Ramp-Hold can store up to six user programs even when the kiln is unplugged. Potters may want to store a program for crystalline glaze, another for a favorite red glaze, etc. Glass artists might want to store several programs for fusing their favorite types of glass and another for slumping.

When you enter Ramp-Hold mode, the first prompt to appear is **USEr.** The **USEr** prompt means, "Select a stored user program for this firing". If you are using Ramp-Hold for the first time, press 1. Then enter num-

ADDENDUM TO COMPLIANCE AGREEMENT WITH MITKEM CORPORATION
DECEMBER 18, 1996
CONTINUED

7. "USDA" soil samples may not be shipped to other facilities unless such facility has a valid permit and compliance agreement for imported soil or a valid compliance agreement for domestic soil. (See Item No. 3 for a list of such facilities)

8. "USDA" soil samples may not be used as a growing medium for plants and for the isolation and/or culture of organisms imported with the soil.

9. It is further agreed that the PPQ Officer may conduct an inspection of the laboratory facility and records concerning soil shipments at any time during normal working hours. All reasonable recommendations of the PPQ Officer to eliminate the spread of plant pests will be followed. Failure to comply with any of the stipulations of this Compliance Agreement may result in its revocation and the facility's authority to handle foreign and domestic regulated soil.

ALL SOIL RESIDUES, THEIR STORAGE CONTAINERS, FILTERS, ETC. SHALL BE TREATED WITH ONE OF THE FOLLOWING SCHEDULES:

Used soil storage containers may be treated by incineration.

DRY HEAT: Soil to be spread in layers 0.5 inches in depth to ensure uniform heat penetration.

TEMPERATURE		EXPOSURE PERIOD
110 - 120.5 C	(230 - 249 F)	16 HOURS *
121 - 154 C	(250 - 309 F)	2 HOURS *
154.5 - 192.5 C	(310 - 379 F)	30 MINUTES *
193 - 220 C	(380 - 429 F)	4 MINUTES *
221 - 232 C	(430 - 450 F)	2 MINUTES *

* Do not start counting time until the mass reaches the required temperature.

STEAM HEAT: 250 F at 15 lbs. pressure for 30 minutes.

Preheat laboratory autoclaves. Do not start counting time until pressure reaches 15 lbs.. Restrict soil depth to 2 inches when treating quantities of soil in trays. Restrict each package weight to 5 pounds or less when treating individual packages. Load with adequate spacing. Large commercial steam facilities which operate at pressures up to 60 lbs. psi will permit treatment of greater soil depth.

For large filter & soil — use 250° for 2 hours

SBL 5/21/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B)

SOP No. 50.0052
Rev 2

	Signature	Date
QA Director:		1/31/08
Lab Director:		2/4/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication
(Method 3550B)

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples using ultrasonic extraction for analysis by EPA SW846 methods SW8081A, SW8082, SW8015 and SW8270C. Discussions include sample extraction, sample cleanup references, and the preparation of standard spiking solutions for the analysis of Total Petroleum Hydrocarbons (TPH), Deisel Range Organics (DRO), pesticide/PCB or semivolatile organic compounds in soil samples. Highly contaminated soils for MA DEP EPH analyses may also use this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. The sample is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using TurboVap or Kuderna-Danish (KD) apparatus followed by Nitrogen blowdown. In addition, this method can be used for Medium/High Concentration Semivolatile samples. A 1-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted once using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection.
- Sample extracts must be analyzed within 40 days of sample extraction.

4.2. Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3. Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1. Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2. Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3. Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1. Ultrasonic Disrupter – pulsing horn type with 375 watts maximum and ¾" standard solid disrupter horn and ⅛" standard tapered microtip probe(medium soils only). Fisher Scientific Sonic Dismembrator Models 500 and 550.

6.2. Sonabox – ultrasonic disrupter box designed to reduce exposure to ultrasonic sound.

6.3. Beakers – 400mL

6.4. 500mL Erlenmeyer flask

6.5. Dessicator

6.6. Balance capable of weighing +/- 0.1 gram.

6.7. Glass syringes for delivering spike and surrogate solutions – 0.25mL, 0.5mL, 1.0mL.

6.8. Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.9. Aluminum Foil (Industrial Grade)

6.10. Anhydrous sodium sulfate: J. T. Baker, ultra resi analyzed grade, granular for drying the samples and sample extracts. Baked at 400°C for four hours.

7. Reagents

The following chemicals including solvents and standards are extensively used in the lab:

7.1. Methylene chloride: J. T. Baker, ultra resi analyzed, to be used for glassware rinsing and sample extraction.

7.2. Methanol: Fisher, Optima, to be used for rinsing glassware and preparing standards.

7.3. Acetone: J.T. Baker, ultra-resi analyzed, to be used for rinsing glassware.

7.4. 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5. Hexane: Fisher, Optima grade, to be used for solvent exchange of samples.

7.6. H_2O, deionized.

7.7. H_2SO_4, concentrated, Fisher, ACS trace metals.

7.8. Surrogate Standards *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9. Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

8. Procedure

8.1. Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1. All **primary standards** received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2. Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is

prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.8** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1mL of the o-Terphenyl primary standards in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains the following compound:

Compound	Concentration(µg/mL)

5-α Androstane	50
o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3. Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard from **section 7.9**, and diluting to 50mL using methanol. The resulting concentration will be 50ug/mL for all SW8270 compounds. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2. The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See **Attachment 1** for a list of the individual compounds and their concentrations.

8.1.3.3. The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.5. The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10mL Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX
where:
SI = Semivolatile intermediate standard
yymmdd = date intermediate standard is prepared,

X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard above, and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following:

Compounds	Concentration(µg/mL)
Diesel fuel	5000

The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.9** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.9** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1.**

A smaller or larger amount may be used to prepare the Lab Control Sample and Matrix Spike Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2. Sample Extraction Procedure:

8.2.1. Low Level Method: Unless Mitkem has documented history that the samples contain high concentration of target analytes (>10,000µg/Kg for the individual analyte) all of the semivolatile samples are prepared using the low-level approach.

Before starting:

- The disrupter has a minimum of 375 watts power and must be inspected periodically to ensure that the ¾ inch tip has not experienced excessive wear.
- The disrupter is tuned each day of use. Follow manufacturer's guidance for tuning procedures (see instructions posted by Sonicators). The tuning is documented in the Sonicator Horn Tuning Logbook, **Figure 4**. After tuning, note in extraction logbook as well.
- Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.1.1. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400mL glass beaker. A 30 gram ± 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopla. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.1.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing, however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.1.3. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes. According to the test method being used, add the following spikes to the samples in beakers:

- Use a syringe to add 1.0mL of the surrogate standard to the Blank, LCS, samples and any MS/MSD sample.

- Use a syringe to add 1.0mL of the LCS/MS standard to the LCS, and any MS/MSD sample.

8.2.1.4 Add 100mL of the 1:1 v/v methylene chloride/acetone mixture to the Blank, LCS, samples, and any MS/MSD for Semivolatile or Pesticide/PCB extraction, OR add 100mL of methylene chloride to the Blank, LCS, samples, and any MS/MSD for TPH/DRO extraction.

8.2.1.5 Place the bottom surface of the ¾ inch tip of the disrupter about ½ inch below the surface of the solvent, but above the solid layer.

8.2.1.6 Extract ultrasonically for 3 minutes, with the output control set at full power and the mode switch on Pulse, using a 50% duty cycle.

8.2.1.7 Remove the beaker from the disrupter. Decant the solvent layer into a 500mL Erlenmeyer flask, leaving the solid in the beaker.

8.2.1.8 Extract the sample two more times with 100mL of solvent and a 3 minute sonication time. Decant the solvent aliquots into the 500mL Erlenmeyer flask. Cover the flask with aluminum foil.

8.3. Semivolatile Medium Level Method: (Also refer to **SOP No. 50.0100** , Method SW846 3570 Microscale Solvent Extraction for another soil extraction option using small sample volumes.)

8.3.1. The sample is mixed to ensure sample homogeneity. As representative as possible, weigh about a 1 gram portion of the sample into a pre-weighed 15mL vial. The weight measurement is recorded to the nearest 0.1 gram.

8.3.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. Mix the sample and anhydrous sodium sulfate to achieve a free flowing mixture.

8.3.3. Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.

8.3.4. Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.3.5. Add 9mL of methylene chloride to the Blank and samples. Add 8mL of methylene chloride to the LCS and any MS/MSD.

8.3.6. Sonicate the sample with the ⅛ inch tapered Microtip for 2 minutes at output control setting at 5 in the continuous mode.

8.3.7. Cap the vial.

8.3.8. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

8.4. The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.

8.5 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:

8.5.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field

samples. Sulfur cleanup will be performed using activated copper powder. Refer to SOP No. 50.0030 Method 3660B Sulfur Cleanup, for details on activiting copper and using it in a sample extract.

8.5.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to SOP No. 50.0031 Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.5.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP No. 50.0032 Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.5.4. Other cleanup methods may be found in SOP numbers 50.0033(Florisil) and 50.0034 (Silica Gel).

8.6 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.7 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.7.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.7.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab

control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1. Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2. Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- An aliquot of the surrogate standard prepared in **section 8.1.2** is added to the Method Blank.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2. Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2. Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1. Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries,

balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4. Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan; Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

Attachments:

Attachment 1: Pesticide LCS/MS List
Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Re-extraction Request
Figure 4: Sonicator Tuning Log

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc. ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES

Date:	Analysis:	Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:

Blank ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	MeCL/Acetone 1:1 Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to Fractionation Date / Analyst	Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments:

Logbook ID: 50.0147-01/08

Surrogate Std. ID:

Matrix Spike/LCS Std. ID:

Reviewed By:_____

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Figure 2
Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonic) 3540C (Soxhlet) 3545(PFE) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH Number:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent Lot # Solvent Name	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst RV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst RV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

	PEST Matrix Spike Std ID:	Vol. Spiked:	
	PCB Matrix Spike Std ID:	Vol. Spiked:	
	PEST Surrogate Spike Std ID:	Vol. Spiked:	
	PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane Lot # / L/L Drip Rate:
	PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot# / Soxhlet Cycle/Hour:
	PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot # / Sonicator Tuned? Yes/No

Logbook ID: 50.0149-02/08

Reviewed By:_____

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-07/07

Reviewed By:_____

Figure 4:
Sonicator Tuning Log

Organic Prep Sonicator Horn Tuning Logbook

See individual Manufacturer Manuals for instructions on tuning, and passing criteria.

Date	Horn ID	Pass	Fail	Comments	Analyst

Logbook ID: 50.0014-05/09

Reviewed by: _____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3570)

SOP No. 50.0100

	Signature	Date
QA Director:		1/31/08
Lab Director:		2/4/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3570)

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil, sediment, tissues, biota and any sample considered solid in nature. Discussions include sample extraction, sample cleanup references, sample concentration technique references and the preparation of standard spiking solutions for the analysis of semivolatile or pesticide/PCB organic compounds in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 2 to 20-gram sample is weighed directly into a 40 or 60-mL VOA vial. The sample is solvent extracted first with acetone. Then it is extract three times with methylene chloride by either a manual shake approach or via rotation or spinning of the sample. For analysis of Pesticides, PCBs and Petroleum samples hexane rather than methylene chloride can be used. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Kuderna-Danish (KD) or TurboVap apparatus.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection
- Samples can be held for one year if they are frozen

- Sample extracts must be analyzed within 40 days of sample extraction

4.2 Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to SOP No. 110.0039 for more detail on sub sampling techniques.

4.3 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

6.1 Rotary extraction apparatus

6.2 40 and 60 mL VOA vials and caps.

6.3 Balance capable of weighing +/- 0.1 gram.

6.4 Glass syringes for delivering spike and surrogate solutions.

7. Reagents

The following chemicals including solvents and standards are extensively used in the lab:

7.1 Methylene chloride: J. T. Baker, ultra resi analyzed, to be used for glassware rinsing and sample extraction.

7.2 Methanol: Fisher, Optima, to be used for rinsing glassware and preparing standards.

7.3 Acetone: J.T. Baker, ultra-resi analyzed, to be used for rinsing glassware.

7.4 Hexane: Fisher, Optima grade, to be used for solvent exchange of samples

7.5 H_2O, Deionized.

7.6 Copper, Fisher, purified. Fine or Granular.

7.7 H_2SO_4, concentrated, Fisher, ACS trace metals.

7.8 Surrogate Standards:

- Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Deisel Range Organics (DRO)/TPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL(Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9 Lab Control Sample and Matrix Spike:

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.

8. Procedure

8.1 Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:
Z = S for Semivolatile, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

8.1.2.1. The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)

Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile working LCS/MS Standard is prepared by combining 2.5mL of Base/Neutral Composite Set primary standard in **Section 7.9**, 2.5mL of Acid Composite Mix primary standard in **Section 7.9** and 2.5mL of Composite Mix #3 primary standard in **Section 7.9** and diluting to 100mL using methanol. All compounds have a final concentration of 50 ug/mL.

8.1.3.2 The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See Attachment 1 for a list of the individual compounds and their concentrations.

8.1.3.3 The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2**.

A smaller or larger amount may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab

and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.2.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

8.2.1. Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.2. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes.

8.2.3. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a 40 mL VOA vial. The sample is weighed into the tared VOA vial. The weight on the sample will vary with the testing procedure and matrix. Tissues usually are weighed in the 3-5 gram range. Soils and sediments are usually in the 5-7 gram range. The data quality objective of the work will dictate if there should be variations from these recommended target weights. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.4. Semivolatile Extraction Procedure:

8.2.4.1. Spike the samples and QC with the appropriate surrogates and spike solutions.

- Use a syringe to add 1.0mL of the Semivolatile surrogate standard to the Blank, LCS, samples and any MS/MSD sample that are in the vials.
- Use a syringe to add 1.0mL of the Semivolatile LCS/MS standard to the LCS and any MS/MSD samples.

8.2.4.2. Add 5-10 mL (7 mL) of acetone to Blank, LCS, samples, and any MS/MSDs. The volume should be enough to **completely cover the sample**. There are instances where samples will appear to absorb the solvent (humic materials for example). In these cases add enough solvent to cover the sample.

8.2.4.3. If sediments are being extracted, added activated copper to aid in the removal of sulfur after the acetone has been added.

8.2.4.4. Cap the vials and shake vigorously for 30 seconds.

8.2.4.5. Allow the samples to settle after agitation.

8.2.4.6. Decant the solvent layer into a 60mL VOA vial, leaving the solid in the 40mL VOA vial.

8.2.4.7. If the samples are extremely wet sediments or tissues, repeat the acetone step from above. At this step, water has been removed from the sample by the acetone.

8.2.4.8. For sediments sulfur is always suspected. Activated copper can be added to the acetone in the 60 mL vials to remove copper.

8.2.4.9. Extract the sample three more times with 5-10 mL (7 mL) of methylene chloride. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.

8.2.5. Procedure for Pesticides, PCBs and Petroleum Hydrocarbon:

8.2.5.1. Spike the samples and QC with the appropriate surrogates and spike solutions.

- Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.
- Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples that is in the 40mL vial.

8.2.5.2. Continue as in **Sections 8.2.4.2 through 8.2.4.8**.

8.2.5.3. Extract the sample three more times with 5-10 mL (7 mL) of Hexane. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.

8.2.5.3.1. For the chlorinated Pesticides and PCBs which are readily extractable, the samples can be extracted by shaking the samples in hexane for three minutes rather than spinning the samples.

8.2.5.4. If the samples contain a petroleum odor, the sample size should be reduced to one to two grams.

8.2.6. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

8.3. Sample Filtration and Concentration: Refer to SOP 50.0054, Extract Filtration and Concentration.

8.4. Extract Cleanup: Refer to SOPs 50.0030 Method 3660B Sulfur Cleanup, 50.0031 Method 3665A Acid Cleanup, 50.0032 Method 3640A GPC Cleanup, 50.0033 Method 3620B Florisil Cleanup, and 50.0034 Method 3630C Silica Gel Cleanup for details on the different cleanup methods available.

8.5. The final extracts are transferred to the Semivolatile lab and documented in the Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.6. Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.6.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.6.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2 Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1 Frequency of LCS:

A LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2 Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS Spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2 Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and

unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3570, Microscale Solvent Extraction, Revision 0, November 2002.

Attachments:
Attachment 1: Pesticide LCS/MS list
Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Re-extraction Request

Attachment 1: Pesticide LCS/MS list

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES, A Division of Spectrum Analytical Inc. ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES

Date:	Analysis:		Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: _____ Soil: 3550B (Sonic) 3540C (Soxhlet)	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:
Blank ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	MeCL/Acetone 1:1 Lot#
H_2SO_4 Lot#	NaOH Lot#	Time/Date Started:	Time/Date Ended:			

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to Fractionation Date / Analyst	Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments _____

Sonicator Tuned: Yes / No
Soxhet Cycle/Hour:_____
Drip Rate_____
Water Bath Temp:_____

Logbook ID: 50.0147-11/07

Reviewed By:_____

Figure 2
Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonic) 3540C (Soxhlet) 3545(PFE) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH Number:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent Lot # Solvent Name	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst RV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst RV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

	PEST Matrix Spike Std ID:	Vol. Spiked:	
	PCB Matrix Spike Std ID:	Vol. Spiked:	
	PEST Surrogate Spike Std ID:	Vol. Spiked:	
	PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane Lot # / L/L Drip Rate:
	PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot# / Soxhlet Cycle/Hour:
	PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot # / Sonicator Tuned? Yes/No

Logbook ID: 50.0149-12/07

Reviewed By:_____

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-07/07

Reviewed By:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8082

SOP No. 60.0003

Rev. 8

	Signature	Date
QA Director:		9/15/08
Lab Director:		9/15/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8082

Rev. 8

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the compounds listed in **Table 1**. This SOP describes the analysis of polychlorinated biphenyls (PCB) as Aroclors in solid or aqueous sample extracts, using a gas chromatograph equipped with an electron capture detector. This SOP gives specific information to perform the analysis according to protocols discussed in USEPA SW846 Test Methods for Evaluating Solid Waste Method 8082A, and Department of Defense Quality System Manual for Environmental Laboratories, Final Version 3. All matrices including ground water, aqueous samples, TCLP and SPLP extracts, petroleum oil, wipes, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require extraction and/or clean-up prior to analysis. **Section 8.2.1** provides the SOP references for sample extraction and clean-up procedures to be used with this analytical procedure. **Section 10** provides the quality control (QC) requirements required by Method 8082. A list of acronyms used in this SOP is included in **Table 2**.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors and Peer analysts** review the logbooks and data generated from this procedure and approve all reported results. The **Data Reviewer** evaluates all laboratory reports for reasonableness of the results. The **Project Manager** reviews the final report and signs the narrative. The **QA**

Director reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

3.1 A sample extract is analyzed by injecting a 1-2µL aliquot into a gas chromatograph (GC) using an auto-sampler. Concentrations of various PCB aroclors are determined by separation of the analytes using a GC equipped with fused silica, open-tubular, megabore columns and electron capture detectors (ECD). Hewlett Packard's Chem-Station (G1034C Version C03.00) is used to handle data acquisition. Target software from ThruPut (Revision 4.14) is used for data reduction and forms generation is through Omega LIMS.

3.2 While SW846 Method 8082 is also applicable to determining polychlorinated biphenyls as congeners, this SOP specifically analyzes PCB as aroclors.

3.3 PCB aroclors are determined using dual-column system with dissimilar phases. The method allows for the option of dual columns joined to a single injection port and individually connected to two ECDs

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by clients and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients provide their own containers. For PCB analysis by USEPA SW846 Method 8082, water samples are collected in 1-liter amber glass bottles with no preservation added to the samples. Solid samples are collected in 8-ounce amber glass containers with no preservation needed. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.

4.2 All sample extracts are stored at 4°C ± 2°C until analyzed.

4.3 Sample extract holding time for PCB analysis by the method is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOP.

4.4 The sample extracts are transferred from Organic Prep Laboratory with all appropriate sample prep information in 2ml auto-sampler vials with Teflon lined crimp cap. All vials should have a meniscus to mark the level of the extracts.

4.5. Samples, sample extracts and standards must be stored separately.

5. Interferences and Potential Problems

5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents, or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve the desired degree of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination. These materials may be removed prior to analysis using Gel Permeation Clean-up (Method 3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and batch analyzing the solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. The solvent rinse should be followed by detergent wash with hot water and rinsed with tap water. Drain the glassware and dry overnight. Store dry glassware in a clean environment. Prior to use, rinse the glassware in methanol and methylene chloride respectively.

5.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCB aroclors. Sulfur contamination may be expected with sediment samples. Method 3660 is suggested for removal of sulfur.

5.5 Waxes, lipids, and other high molecular weight materials may interfere with the analysis of PCB aroclors and can be removed by Gel-Permeation Clean-up (GPC) (Method 3640). Co-eluting chlorophenols can be removed by florisil clean-up (Method 3620).

6. Equipment and Apparatus

6.1 Gas Chromatograph/Electron Capture Detector

There are 5 GC/ECD in the laboratory. Three of the instruments have similar configurations and are labeled as E1, E2 and E3. The other two are GC/μ-ECD are labeled as E4 and E5. GC E1, E2 and E3 – Hewlett Packard (HP) Model 5890 Series II with electron Pressure Control. GC E4 and E5 - HP Model 6890 Series II with u-Electron Pressure Control. All five GC's are temperature programmable instruments with single injection port and dual electron capture detectors, and are equipped with HP Model 7673A Auto-sampler. A single gooseneck splitless injection liner is used in the injection port and connected to a piece (up to a 5m long) of uncoated megabore column (0.53mm id) which serves as a guard column. A guard column is connected to the tee split. Each of two columns is connected to the split tee also. HP 3365 Chemstation software is used in conjunction with EnviroQuant software to handle data acquisition and processing. All GC's are interfaced to the network workstation. LIMS software is used for reports.

6.1.1 HP Vectra PC.

6.1.2 HP 3365, EnviroQuant and Target software.

6.1.3 HP Model 7673A auto-sampler.

6.1.4 Three pairs of chromatographic columns are intensively used in the laboratory:

Pair 1 DB-1701 column (J & W Scientific) 30 m x 0.53mm id (1.0 μm film thickness) and DB-608 column (J & W Scientific) a 30 m x 0.53mm id (0.83 μm film thickness)

Pair 2 RTX-CLPesticides (Restek) 30 m x 0.53 mm id (0.5 um film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.53 mm id (0.42 um film thickness)

Pair 3 RTX-CLPesticides (Restek) 30 m x 0.32 mm id (0.5 um film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.32 mm id(0.42 um film thickness).

6.1.5 Gooseneck splitless injection liner, Cat #20799 from Restek or equivalent

6.1.6 Uncoated megabore guard column (0.53 mm id, up to 5 m long). Cat # 10028 from Restek or equivalent.

6.1.7 Universal "Y" Press-tight tee split Cat # 20406 from Restek or equivalent.

6.2 Glassware:

6.2.1 Class "A" volumetric flasks:
10 ml, 25 ml, 50 ml, 100 ml, and 250 ml.

6.2.2 Syringes:
10 µl, 25 µl, 50 µl, 100 µl, 500 µl, 1 ml, 2.5 ml (accuracy to ± 1% per vendor's specification).

7. Reagents and Standards.

7.1 n-Hexane: Fisher Optima grade.

7.2 The standards used in the method are discussed below. Please note that standards from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials. The laboratory will at all time archive or have on order one complete set of un-opened ampulated standards.

7.3 The list of primary (ampulated) calibration standards are obtained from Restek:

- Aroclor 1016 and 1260 (Cat. # 32039) at 1000ug/mL.
- Aroclor 1221 (Cat.# 32007) at 1000ug/mL.
- Aroclor 1232 (Cat. # 32008) at 1000ug/mL.
- Aroclor 1242 (Cat. # 32009) at 1000ug/mL.
- Aroclor 1248 (Cat. # 32010) at 1000ug/mL.
- Aroclor 1254 (Cat. # 32011) at 1000ug/mL.
- Tetrachloro-m-xylene (TCX) (Cat.# 32027) at 200 ug/mL.
- Decachlorobiphenyl (DCB), (Cat.# 32029) at 200 ug/mL

7.4 The list of second source standards are obtained from Supelco:

- Aroclor 1016 (Cat. # 4-8097) at 1000ug/mL.
- Aroclor 1221 (Cat. # 4-8098) at 1000ug/mL.
- Aroclor 1232 (Cat. # 4-4805) at 1000ug/mL.
- Aroclor 1242 (Cat. # 4-4806) at 1000ug/mL.
- Aroclor 1248 (Cat. # 4-4807) at 1000ug/mL.
- Aroclor 1254 (Cat. # 4-4808) at 1000ug/mL.
- Aroclor 1260 (Cat. # 4-4809) at 1000ug/mL.

7.5 All of the above primary standards are logged into the Primary Pesticide Standard Logbook and labeled as **PP*yymmddX***

Where: PP = Pesticides/PCB primary standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in alphabetical order.

The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. All primary standards are stored according to manufacturer's recommendation. All vials containing primary standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability.

7.6 Intermediate Standards.

All intermediate standards are labeled **PI*yymmddX*** where:

PI = Pesticide Intermediate Standard.
yymmdd = date the intermediate standard is prepared.
X = the order that the intermediate standard is prepared on that date in alphabetical order.

Smaller or larger volumes may be used and the final volume adjusted to keep the concentration the same.

All intermediate standards are stored in amber glass bottles under refrigeration at 4°C or below. All standards are stored separately from samples and sample extracts

All intermediate standard solutions must be prepared every six months, or sooner, if the solution has degraded or concentrated.

All of the appropriate standard preparations are recorded in the Pesticide/PCB Intermediate Standard Preparation logbook.

Analyst must allow all intermediate standard solutions to equilibrate to room temperature before use.

7.6.1 Intermediate Pesticide/PCB Surrogate Mix:

The intermediate stock standard is made by diluting 0.5mL of stock TCX and 1.0 mL of stock DCB and diluting to 50 mL with hexane. Intermediate Pesticide/PCB Surrogate Mix contains TCX at 2 ug/mL and DCB at 4ug/mL.

7.6.2 Intermediate AR1660 (AR1016+AR1260) Stock Solution:

The two Aroclors –Aroclor1016 and Aroclor 1260 have different chromatographic patterns and can be combined for analysis. When the two Aroclors are combined, they are labeled as Aroclor 1660.

The mixture of the two Aroclors is prepared by diluting 1mL of primary Aroclor standard at 1000ug/mL (AR 1016 + AR1260) Mix into 100mLs of hexane.

Concentration of the intermediate AR1660 standard solution is10ug/mL

7.6.3 Aroclors 1221, 1232, 1242, 1248 and 1254 Intermediate Stock Solutions:

Intermediate stock solutions of each of the aroclors are prepared by diluting 1mL of the primary standards (at 1000ug/mL concentration) to 100mLs using hexane. There will be one intermediate solution for each of the aroclors listed above, each at a concentration of 10µg/mL.

Please note that the following preparation procedures pertain to the use of the primary stock standards listed in **Sections 7.3** and **7.4**. Different preparation schemes are needed if different stocks are used.

7.7 Working Standards for Aroclor 1221, 1232, 1242,1248, 1254 and1660.

The working 5 levels calibration standards for each of the individual Aroclors is prepared as follows:

Level 5: 8mL of the intermediate stock Aroclor prepared **in Section 7.6.2**. and 2mL of the intermediate surrogate standard prepared in **Section 7.6.1**.are diluted to 50mL using hexane.

Note: The preparation volumes could be adjusted to make smaller or larger volume of standards as long as the final working concentrations are the same.

Level 4: 25mL of level 5 are diluted to 50mL using hexane.

Level 3: 25mL of level 4 are diluted to 50mL in hexane

Level 2: 25mL of level 3 standard is diluted to 50mL using hexane.

Level 1: 25mL of the level 2 standard are diluted to 50mL using hexane.

7.7.1 The 5 levels of Aroclor 1660 standards contain the following (concentration in ug/mL):

	Aroclor 1016	Aroclor 1260	TCMX	DCB
Level 1	0.1	0.1	0.005	0.01
Level 2	0.2	0.2	0.01	0.02
Level 3	0.4	0.4	0.02	0.04
Level 4	0.8	0.8	0.04	0.08
Level 5	1.6	1.6	0.08	0.16

7.7.2 The working 5 level of each of Aroclors contain the following (concentration in ug/ml):

	Aroclor	TCMX	DCB
Level 1	0.1	0.005	0.01
Level 2	0.2	0.01	0.02
Level 3	0.4	0.02	0.04
Level 4	0.8	0.04	0.08
Level 5	1.6	0.08	0.16

7.7.3 Second source standards:

The second source standards are obtained from Supelco. All Aroclors are individually prepared at 0.4 ug/mL.

Please note that surrogate standards may or may not be added to the second source standards.

All of the above working standards are logged into the Pesticide Working Standard Logbook and labeled as PW*yymmddX*

where: PW = Pesticides/PCB working standard
yymmdd = date the standard is prepared
X = the order the standard is prepared on that date, in alphabetical order

All working standards are stored in amber containers under refrigeration at 4°C or below. The standards are stored in separate location from the samples and/or extracts to minimize cross contamination.

The expiration date for the standards is six months from the date of preparation or whenever the primary standard expires, whichever comes first.

8. Procedure

8.1 Preparation – The methods in USEPA SW-846 for sample extraction are as follows:

Method 3510 (SOP# 50.0022) extracts aqueous samples for water-insoluble and slight water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

Method 3520 (SOP# 50.0025) extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0019) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using 1:1 (v/v) methylene chloride/acetone in a soxhlet extraction.

Method 3550 (SOP# 50.0016) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using ultrasonic extraction in 1:1 (v/v) methylene chloride/acetone.

Method 3570 (SOP# 50.0100) is the extraction of Pesticides in soil, sediment, tissues, biota and any sample considered solid. A 2-20gram sample is solvent extracted first with acetone, and then with hexane by either a manual shake approach or via rotation or spinning of the sample.

In addition, clean-up procedures are employed to remove co-eluting interferences. The lab uses GPC clean-up (Method 3640) and sulfur clean-up (Method 3660).

8.2 Instrument conditions

The GC operating conditions are as follows:

Carrier Gas	Helium (99.999%)
Column Flow	5-10 mL/min., independent of temperature
Make-up Gas	5% methane/95% argon (P-5)
Make-up Gas Flow	80-100 mL/min.

Injector	Split-less
Injector Temperature	200°C
Initial GC Temperature	120-170°C
Initial GC Hold	1 min.
Temperature Ramp	6-8°C/min
Final Temperature	265-300°C
Final GC Hold	2-15 min.
Detector Temperature	300°C

8.2.1 Optimize GC operating conditions for analytes separation and sensitivity. Once optimized the same conditions must be used for analysis of all standards, samples, blanks and MS/, MSD.

The auto-sampler makes 2µl injection for the analysis of all standards and sample extracts.

8.2.2 Preventative Maintenance.

The injector septum is replaced every time the instrument is set up to perform a sequence of analyses when a leak develops, or when initial and/or continuing calibrations fail to meet the method requirements.

The injection liner is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops or when initial and/or continuing calibrations fail to meet the method requirements. The gold seal will be replaced and the columns will be trimmed every time before a new calibration is run.

The column will be replaced if standard chromatograms show excessive peak tailing or initial and/or continuing calibrations repeatedly fail to meet the method requirements.

8.2.3 Major instrument maintenance must be documented in the LIMS maintenance logbook, regular (daily) maintenance can be documented in Instrument Run Logbook.

8.2.4 Initial calibration is performed by analyzing 5 level standards of the Aroclor 1660, and at least one level (midpoint) of the following:

1 Aroclor 1221
2 Aroclor 1232
3. Aroclor 1242
4. Aroclor 1248
5. Aroclor 1254
6. Aroclor 1660

All analytical runs need to be documented in the appropriate Instrument Run Log. In addition to listing the data file, the associated working standard ID and standard name should be noted.

The chromatograms for each of the target Aroclor standards are presented in **figures 1 - 7** for both the RTXCLPest and RTXCLPest II columns.

8.2.5 From the initial calibration, the following parameters are determined:

8.2.5.1 Retention time and retention time window:

The retention time window is established by evaluating the retention time of the Aroclor 1260 standards (at 1ug/mL) that were analyzed over a 72-hour interval. Please note that the laboratory does not need to perform retention time window studies for the other Aroclors.

From the standard analysis, three peaks are selected for each of Aroclor 1016 and Aroclor 1260. The retention time windows for the six peaks are determined as ± 3 times the standard deviation from the 72-hour interval. If the standard deviation is zero, a default standard deviation of 0.01 minutes will be used, per SW-846 8000C, Section 11.6.3.

The retention time windows are established by centering the ±3 standard deviation around the retention time of the six peaks in the mid level standard that was analyzed as part of the 5 level initial calibration.

It is Mitkem's experience that the GC retention times operating under EPC conditions is very tight. Using the above approach in determining retention time windows at times has resulted in extremely narrow retention time windows. This may result in false negatives due to slight retention time shifts, especially when sample extracts are subjected to co-eluting interferences. In such cases, the SW-846 default standard deviations will be used.

For the surrogate standards, the retention time window is established using the same approach as the multi-component PCBs. The above retention time window criteria also apply.

8.2.5.2 Mitkem chooses to use external quantitation for instrument calibration and sample quantitation. The following equation is used to calculate the calibration factor (CF):

$$CF = \frac{\text{Peak area (or height) of standard}}{\text{Mass injected (ng)}}$$

The above equation is used to determine the calibration factor for both the single component and multi-component standards. Please note that either peak area or peak height can be used for the calculation.

The lab chooses to use peak height for single component analytes such as the surrogate compounds and for the multi-component PCB.

In calibrating for the Aroclors, the laboratory selects three peaks that are characteristic of each of the Aroclors. The analyst will choose peaks in each Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Also, it is preferable that one of the three peaks selected is unique to that Aroclor.

8.2.5.3 Linearity - linearity is used to evaluate the dynamic range of the calibration factor for each of the single component surrogate standards. Linearity, as measured by the % Relative Standard Deviation (RSD) is calculated using:

$$\% \text{ RSD} = \frac{SD_{CF}}{CF_{av}} \times 100$$

Where: $SD_{CF} = \sqrt{\sum (CF_i - CF_{av})^2 / (n-1)}$
CF_{av} = average calibration factor
CF_i = calibration factor
n = total number of values = 5

Please note that there are three sets of calibration factors for each of the Aroclors.

8.2.5.4 <u>%RSD</u> Acceptance criteria for linearity:

The acceptance limit for linearity check is that the % RSD for the <u>individual Aroclor calibration factors must</u> be less than or equal to 20%. If this condition is met, the instrument is determined to be linear within the calibration range.

Given the large number of <u>individual compound</u> calibration factors, it is likely that some Aroclors may exceed the 20%

acceptance limit. For Aroclors that exceeded the 20% limit, the linearity check is still acceptable if the following least square regression conditions are met:

8.2.5.5 Alternate Least Square Regression criteria for Linearity:

The analyst may employ a regression equation for the Aroclor(s) that do(es) not pass using the earlier approach. The regression will produce the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where y = instrument response (peak area or height)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is not included as the Sixth calibration point and that the above equation is not forced through the origin. The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$ ($r^2 \geq .0.990$)

8.2.6 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. The calibration is performed by analyzing standard Aroclor 1660 standard prepared in **Section 8.1.8**. The acceptance criteria are as follows:

The calculated value of the analyte in the ICV should be 80 – 120% of the expected value. If the ICV does not meet the criteria, see corrective action guidelines in **Attachment 1** of this SOP. Results of this evaluation should be documented in the Instrument run log.

If the above criteria are not met, the analyst must evaluate the integrity of the primary and confirmation standards. If needed, re-preparation and re-analysis of the initial calibration is required.

8.2.7 Initial calibration acceptance criteria must be met before any sample, blanks or LCS are to be analyzed.

8.2.8 Corrective Action for Initial Calibration – see **Attachment 1** for corrective action guidelines and documentation.

8.2.9 Original initial calibration raw data must be archived in the company organics analysis calibration (OCAL) database. See Mitkem SOP # 10.0009 (Application Xtender) for scanned archive information.

8.3 Continuing Calibration Verification and Sample Analysis – Method 8082 requires the analysis of Aroclors 1016/1260 as the continuing calibration verification (CCV) every time samples are to be analyzed to ensure that the GC/ECD system continues to meet instrument sensitivity and linearity requirements. Mitkem may also analyze Aroclors 1242, 1248 and 1254 as part of the calibration verification

8.3.1 Frequency of Continuing Calibration (CCV)- The CCV must be performed if the instrument has been idle for more than 12 hours. A continuing calibration standard must also be injected at intervals of not less than once every twenty field samples (the method recommends the frequency to be once every ten samples, to minimize reanalyses due to unacceptable CCVs) and at the end of the analysis sequence.

DoD requires CCV standards must be injected at intervals of not less than once every ten field samples.

8.4.1 The continuing calibration is performed using the Level 3 (0.4 ug/ml) Aroclor 1660 standard (as well as Aroclors 1242, 1248 and 1254 where appropriate). While the method suggests alternating the use of high and low calibration mixtures, Mitkem performs the continuing calibration using the mid-point standard. When the additional Aroclors 1242, 1248 and 1254 are analyzed, they will also be evaluated using the same approach as Aroclors 1016/1260.

8.3.2 Evaluation of the Calibration Verification:

8.3.2.1 *% Difference:* Calculate the % difference between the continuing calibration CF and those from the most recent initial calibration.

The % difference is determined as follow:

$$\% \text{ Difference} = \frac{CF_c - CF_i}{CF_i} \times 100$$

In which:

CF_c = calibration factor from continuing calibration
CF_i = mean calibration factor from the most recent initial calibration

8.3.2.2 *% Drift:* Use % drift when using linear regression calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

In which: $Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.3.2.3 *% Difference (or % Drift)* between the continuing calibration factor and that of the initial calibration should be no greater than ± 20% at least on one column. If this criterion is met then the calibration has been verified and sample analysis can proceed. The analyst should mark whether the CCV passed on one or both columns in the instrument logbook. Checkmarks or the term "OK" are acceptable entries .

8.3.2.4 Comparison of the retention time window: the retention time of each aroclor and surrogate peak in the calibration standards should fall within the retention time window established from the initial calibration. Due to the need to perform routine column maintenance (clipping off a small length of the guard and/or analytical columns), the retention time might be shortened. In such cases, either the RT window is adjusted to agree with the calibration verification retention times or new retention times are established using the calibration verification standard.

8.3.3 Corrective Action for opening CCV- The %D (or % Drift) between the individual compound calibration verification factors and those from the initial calibration should be no greater than ±20% on at least one column. If this criterion is exceeded for any peak, the calibration has not been verified.

If the continuing calibration does not meet the criteria, a second injection of the calibration verification is allowed. If the second injection fails, the analytical sequence is stopped. Corrective actions must be performed. This may include preparing new standards, performing inlet maintenance and/or GC column maintenance. If the response of the analyte is still not within the above criteria, then a new initial calibration must be performed.

8.3.4 Corrective Action for closing CCV: When a calibration verification standard fails to meet the QC criteria on both columns, all samples that were injected after the last compliant standard must be evaluated to ensure the data is valid. The analyte(s) which fail(s) the 20%D must be addressed if the analyte affected is one that was detected in project sample(s). The analyst needs to note all QC issues in the instrument run

log and on all associated project data review checklists. This information needs to be addressed in the associated project(s) narrative. See **Attachment 1** for corrective action guidelines and documentation.

8.3.4.1 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**), and the analyte **was not** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, the group of samples is deemed acceptable and does not require re-analysis.

8.3.4.2 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on both columns, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, then re-injection of all samples are required to ensure accurate quantitation.

8.3.4.3 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on one column, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards,and the associated samples are detects, the reported values must come from the compliant column, even if this is the higher result.

8.3.4.4 If the non-compliant standard analyzed **after** a group of samples exhibits a lower response for an analyte that is greater than 20% D (**negative bias**), then re-injection of all samples are required to ensure accurate quantitation.

Sample injections may continue as long as the calibration verification standards meet the instrument QC requirements.

All standard analyses are to be documented in the appropriate Instrument Run Log with associated working standard IDs.

8.3.5 The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Target software. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample. Both retention time and pattern are important when determining PCBs in a sample.

It is important that if Aroclors are detected in the samples, the analysis of these sample extracts should be accompanied by the appropriate Aroclor calibration verification standard as part of the analytical sequence. If this is not done, the sample extracts have to be re-injected with the appropriate Aroclor standards to ensure the PCB pattern is similar.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. In these instances, the analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclors.

8.3.6 The concentration of the target compounds and surrogates are calculated separately for each of the three Aroclor peaks for both columns using the following equation.

For aqueous samples, concentration *ug/L* = $\frac{(Ax)\ (Vt)\ (DF)}{(CF)\ (Vo)\ (Vi)}$

For soil samples, concentration *ug/kg* = $\frac{(Ax)\ (Vt)\ (DF)}{(CF)\ (Ws)\ (Vi)\ (S)}$

in which: Ax = area/height of the peak for the compound to be determined

CF = calibration factor
Vo = volume of water extracted, in mL
Ws = weight of soil sample, in gram
Vi = volume of extract injected, in µL
Vt = volume of extract, in µL
DF = dilution factor
S = solid content, expressed in decimal

For the multi-component Aroclors, the analyte concentration is calculated by taking the average of the concentration determined for each of the three peaks selected for that analyte.

8.3.7 Manual integration will be performed if needed and documented according to the current revision of SOP Number 110.0008. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. The analyst will further initial and date the manual integration with the proper reason code per SOP No.110.0008.

8.3.8 The concentrations of the surrogate analytes are calculated and reported separately for each of the two columns. The acceptance limits for the recoveries are discussed in **Section 10**.

8.3.9 The analyte recoveries of the LCS and matrix spike compounds are determined and reported. The acceptance limits for the recoveries are discussed in **Section 10**.

8.4 Analytical sequences are summarized as follows:

Order	Sequence	Comment
1	AR1232L3	
2	AR1221L3	
3	AR1242L1	Level 3 only for AR1232 and AR1221 are run unless this Aroclor is detected in samples.
4	AR1242L2	
5	AR1242L3	
6	AR1242L4	
7	AR1242L5	
8	AR1248L1	
9	AR1248L2	
10	AR1248L3	
11	AR1248L4	
12	AR1248L5	
13	AR1254L1	
14	AR1254L2	
15	AR1254L3	
16	AR1254L4	
17	AR1254L5	
18	AR1660L1	
19	AR1660L2	
20	AR1660L3	
21	AR1660L4	
22	AR1660L5	
22	AR1660SS	Second source ICV

23	BIBLK01	Instrument blank
24 25 26	Blank LCS Sample extracts	
27 28 29 30	AR1242M01 AR1248M01 AR1254M01 **AR1660M01**	calibration verification
31	BIBLK02	
32	Sample extracts	
33 34 35 36	AR1242M02 AR1248M02 AR1254M02 **AR1660M02**	
37	BIBLK03	
38…	Sample extracts…	Sequence continues as the same as above as long as calibration verifications meet the acceptance criteria.

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight for solid samples.

Results are reported to two significant figures using the USEPA guidelines in rounding up or down. For solid samples, results are reported in dry weight unless otherwise specified.

For 8082 analyses, PCB aroclors are reported if the determined concentration is above the project reporting limits on both columns. No J flagged results are reported.

9.2 Soil concentrations are calculated using dry weight basis. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids.

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
WW = Sample weight (g) before drying

9.3 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100\% \times (SSR-SR)/(SA)$$

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.4 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100\%$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks. A method blank is an aliquot of a clean reference matrix (reagent water for water samples, or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. A Method blank is prepared and analyzed with every batch of 20 samples or less. It is used to determine the level of contamination associated with the analytical processing and analysis of the samples. The Method blank MUST be analyzed on the same instrument as the associated samples.

See **Attachment 1** for additional corrective action.

10.2.1 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.

10.2.2 The concentration of target compounds in the method blank must be ≤ to ½ CRDL or RL.

10.2.3 Corrective action for method blank contamination involves determining the source of the contamination and possibly re-extracting the entire batch.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a volume or weight of a clean reference matrix (organic-free water or Ottawa Sand) that is spiked with Aroclors 1016 and 1260 and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 The recovery of the surrogates must be within the calculated acceptance limits discussed **Section 10.6**.

10.3.2 The LCS recovery is evaluated against the established in-house limits. Any analyte/analytes not meeting the criteria require corrective action. See **Attachment 1**. For DoD projects, see **Attachment 2**, DoD Tables D-14 and 15.

10.3.3 Advisory RPD limits are set at 30% when duplicate LCSs are performed.

Any sample(s) that is/are associated with a non-compliant LCS may require re-extraction and re-analysis. See **Attachment 1** for corrective action guidelines.

10.4 Duplicate Matrix Spikes –Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix at a frequency of one set per 20 samples.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and one duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform a matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above.

10.4.2 Control limits for Method 8082 projects are the same as discussed in **Sections 10.3.2 and 10.3.3.** DoD requires 30% RPD for all matrices. See **Attachment 2**, DoD Table B-2.

Depending on sample matrix, chromatographic based analysis such as Method 8082 is susceptible to co-eluting interferences resulted in high biased values. Thus, the control limits for the duplicate matrix spikes should be used as guidelines. The determination of outliers should be addressed in the project narrative to advise the data users of potential matrix related interferences.

10.5 Re-analysis at dilution - Any target compounds that are determined above the instrument calibration range will require reanalysis at dilution. A notation for the need to perform a dilution next to the sample run in the instrument logbook is needed to document the results. An entry such as " rerun at 4x " is acceptable.

The dilution is performed by taking an aliquot of the extract and diluting it to a pre-determined volume using hexane. The analyst should not over-dilute the extract. The analytes that trigger the need for dilution should be determined in the diluted analysis at a concentration above that of the midpoint calibration standard.

10.5.1 When reporting diluted results the following guidelines should be followed. If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis may be reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the DL suffix appended to both the client and sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and all compounds detected in the more diluted (DL) analysis are qualified with the "D" flag.

10.5.2 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument, the initial analysis may be performed at dilution. If this analysis is acceptable (compounds at or above the mid-point calibration) then a less dilute analysis is not required. The sample ID is not changed by adding a DL suffix but the 'initial analysis at dilution' is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

10.5.3 If the initial analysis fails QC criteria, it may be reported if specified by the project or client. If only the dilution is reported, the DL suffix is not added and the dilution is noted on the data review checklist submitted with the data for review as above.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks, MS/MSD and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1 Acceptance criteria for surrogate recovery.

For 8082 projects the percent recovery of each of the surrogate compounds in method blanks and LCS must be within the in-house acceptance criteria window. The current in-house acceptance criteria for each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS are listed under the option SPEC for the Omega LIMS Testcode.

In-house limits may be lab generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab generated limits are derived, these will be updated annually.

Surrogate spikes in matrix specific samples that fail to meet the in-house limits would indicate potential matrix effect. In general, high recovery of surrogates due to co-eluting interferences is acceptable. Low recovery of surrogates would indicate potential matrix related problems or one related to the extraction process. The analyst should confirm the sample volume extracted and the final extract volume to verify the calculations. If applicable, the analyst should consider additional cleanup procedures and re-analysis. If no appropriate cleanup procedure could be identified or re-analysis after additional cleanups failed to achieve the limit, this should be noted on the review checklist and also addressed in the narrative.

If surrogate recoveries do not meet recovery criteria see **Attachment 1** for corrective action

10.7 Annually, MDL studies are conducted to establish the limit of detection applicable to this method. Please refer to the SOP No. 80.0005 for more detail. An MDL verification at approximately 2XMDL will be analyzed after the study. MDL verification may be analyzed quarterly in lieu of the annual study.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, is reviewed for technical accuracy and completeness by the analyst. The analyst initiates a project data review checklist and documents all comments regarding analysis there. Sample preparation logs, notebooks, and instrument logs are reviewed and signed by the laboratory supervisor. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 All raw data is peer reviewed at the computer/Target level by another analyst or the lab supervisor prior to final form generation. Analysts generate all hard copy raw data and upload electronic data files to the OMEGA LIMS for reporting. Raw Data, including all support data (such as data review checklist, run-logs, work-order sheets, SDG summaries…) are brought to the data reporting department for assembly. After assembly, all data are reviewed by senior personnel (data reviewers) for quality control and completeness dependent on project specific requirements.

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. See **Attachment 1** for routine corrective action guidelines and documentation.

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedures SOP No. 80.0007.

13. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible. Spent vials and ampules are disposed of into a red metal drum in the Semivolatile Laboratory. See SOP No. 30.0024, Sample and Waste Disposal for more detail.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention; Waste Management; Definitions and Acronyms

See Sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency. Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Method 8082A, SW-846 Test Methods for Evaluating Solid Wastes, Final Update IV, Feb 2007.
2. "Methods Compendium for Inorganic and Organic Methods," United States Army Corps of Engineers, Appendix I, 2001.
3. "Shell for Chemical Analytical Requirements," United States Army Corps of Engineers, Appendix H, 1997 including addendum dated 1 February 2001.
4. Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

Attachments:

1. **Table 1**: List of Target Analytes.
2. **Table 2**: List of Acronyms.
3. **Figure 1**: Aroclor 1660 Standard Chromatogram and Quantitation Report.
4. **Figure 2**: Aroclor 1221 Standard Chromatogram and Quantitation Report.

5. **Figure 3**: Aroclor 1232 Standard Chromatogram and Quantitation Report
6. **Figure 4**: Aroclor 1242 Standard Chromatogram and Quantitation Report
7. **Figure 5**: Aroclor 1248 Standard Chromatogram and Quantitation Report
8. **Figure 6**: Aroclor 1254 Standard Chromatogram and Quantitation Report
9. **Attachment 1**: Corrective Action Examples and Documentation Tables
10. **Attachment 2:** DoD QC Requirements: SW 846 DoD-B, Tables B-2, D-14, D-15.

Table 1

List of Target Analytes for Method 8082

Aroclor	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

Table 2

List of Acronyms

PCB	Polychlorinated Biphenyl
TCLP	Toxicity Characteristic of the Leaching Procedure
SPLP	Synthetic Precipitate Leaching Procedure
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate
GC	Gas chromatograph
ECD	Electron capture detector
GPC	Gel Permeation chromatography
CF	Calibration factor
RSD	Relative standard deviation
RT	Retention Time
EPC	Electron pressure controller
USACE	US Army Corp. of Engineers
AFCEE	Air Force Center of Environmental Excellence
CLP	US EPA Contract Laboratory Program
DoD	Department of Defense

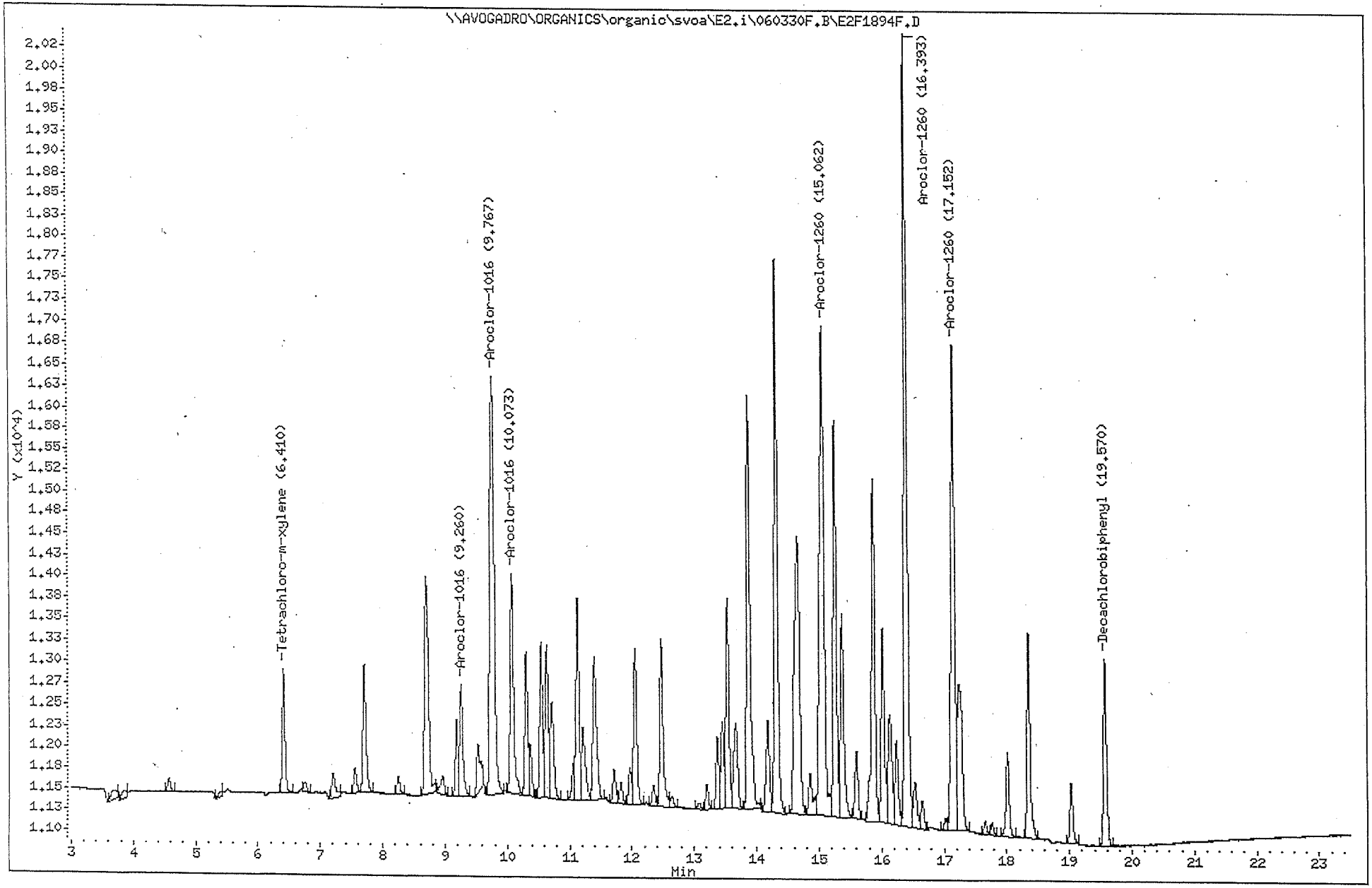
Figure 1

Aroclor 1660 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D
Date : 31-MAR-2006 02:31
Client ID: AR1660FL1
Sample Info: AR1660FL1,AR1660FL1,,ar1660.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

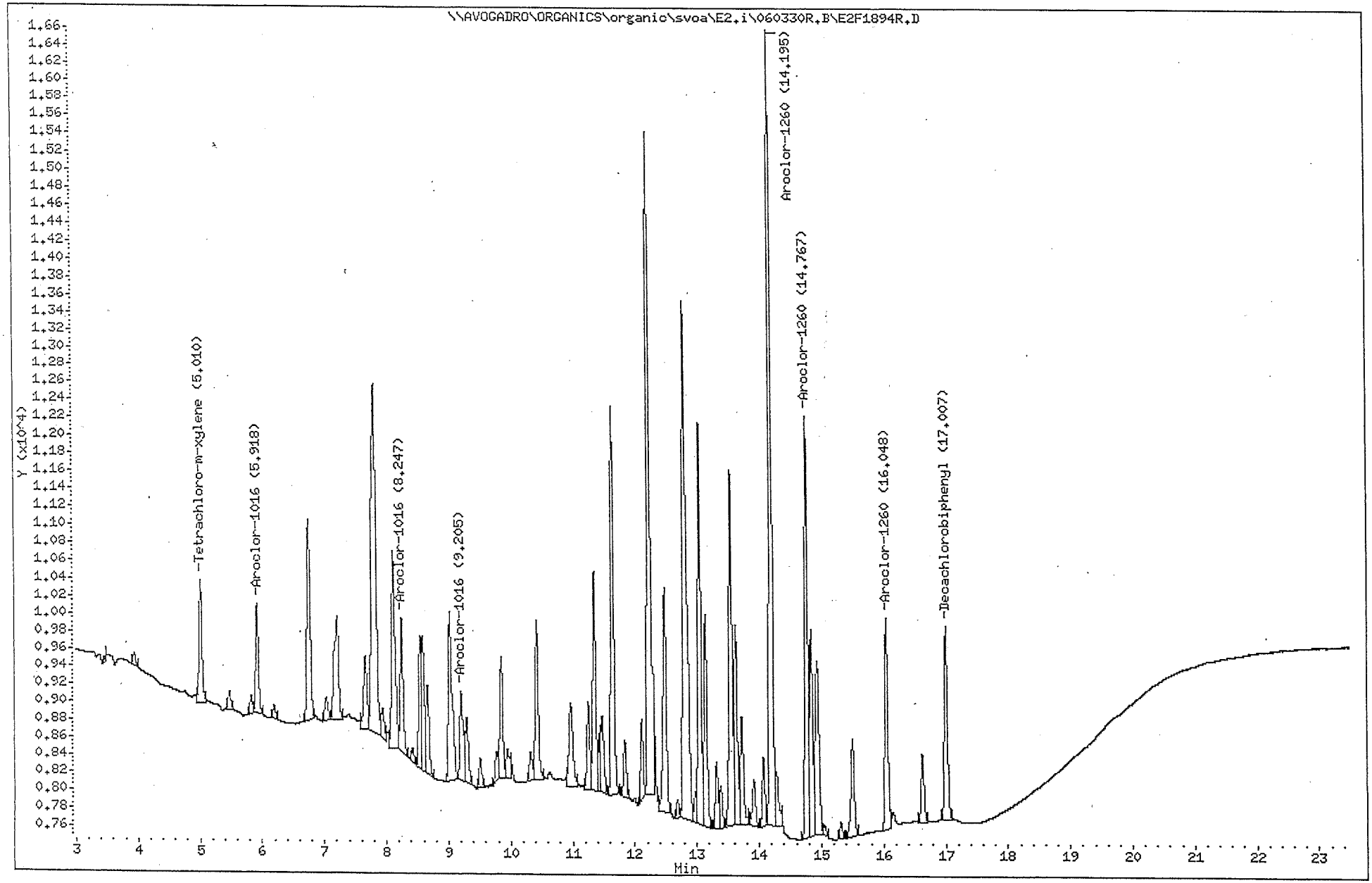
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D
Date : 31-MAR-2006 02:31
Client ID: AR1660FL1
Sample Info: AR1660FL1,AR1660FL1,,ar1660.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1894F.D
Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D
Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
Inj Date : 31-MAR-2006 02:31
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 18 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1660.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1471	0.00200	0.0022			(a)
4 Aroclor-1016					CAS #: 12674-11-2			
9.26	9.26	0.000	1314	0.10000	0.12	80.00- 120.00	100.00	(a)
9.77	9.77	0.000	4921	0.10000	0.13	327.41- 367.41	374.51	
10.1	10.1	0.000	2590	0.10000	0.12	166.80- 206.80	197.11	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2221	0.00200	0.0026			(a)
8 Aroclor-1260					CAS #: 11096-82-5			
15.1	15.1	0.000	5765	0.10000	0.13	80.00- 120.00	100.00	(a)

3/31/06

Data File: E2F1894F.D
Report Date: 31-Mar-2006 07:55

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
8 Aroclor-1260 (continued)							
16.4	16.4	0.000	9365	0.10000	0.12	151.74- 191.74	162.45
17.2	17.2	0.000	5721	0.10000	0.12	86.33- 126.33	99.24
		Average of Peak Amounts =			0.12		

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: E2F1894R.D
Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D
Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
Inj Date : 31-MAR-2006 02:31
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 18 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1660.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1405	0.00200	0.0025			(a)
6 Aroclor-1016					CAS #: 12674-11-2			
5.92	5.91	0.010	1254	0.10000	0.12	80.00- 120.00	100.00	(a)
8.25	8.24	0.010	1526	0.10000	0.12	112.38- 152.38	121.69	
9.21	9.20	0.010	1023	0.10000	0.12	72.72- 112.72	81.58	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2198	0.00200				(a)
8 Aroclor-1260					CAS #: 11096-82-5			
14.2	14.2	0.000	8966	0.10000	0.13	80.00- 120.00	100.00	(a)

3/31/06

Data File: E2F1894R.D
Report Date: 31-Mar-2006 07:55

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
8 Aroclor-1260 (continued)							
14.8	14.8	0.000	4745	0.10000	0.13	32.61- 72.61	52.92
16.0	16.0	0.000	2397	0.10000	0.12	8.08- 48.08	26.73
		Average of Peak Amounts =			0.13		

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

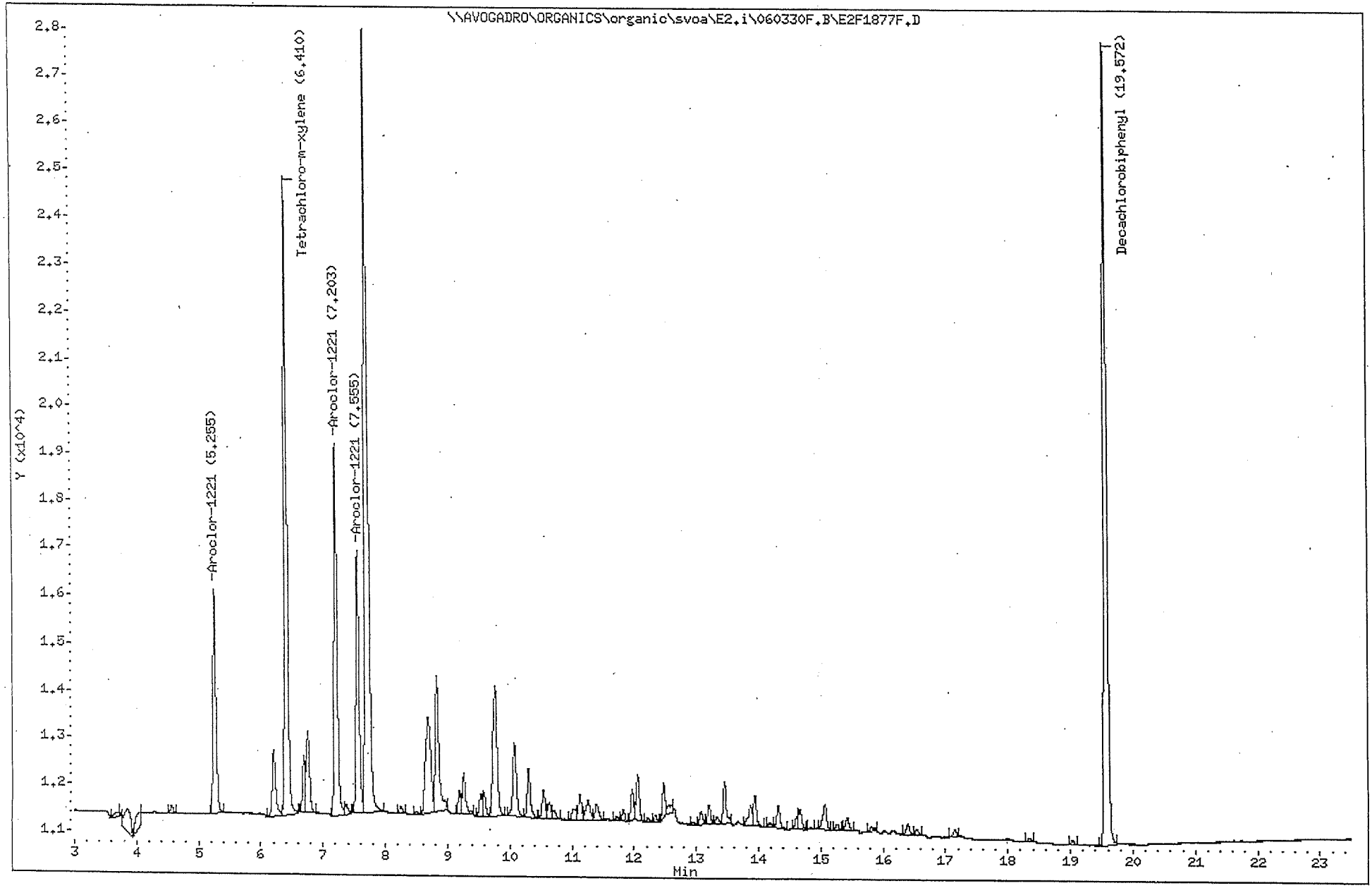
Figure 2

Aroclor 1221 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D
Date : 30-MAR-2006 18:58
Client ID: AR1221FL3
Sample Info: AR1221FL3,AR1221FL3,,ar1221.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

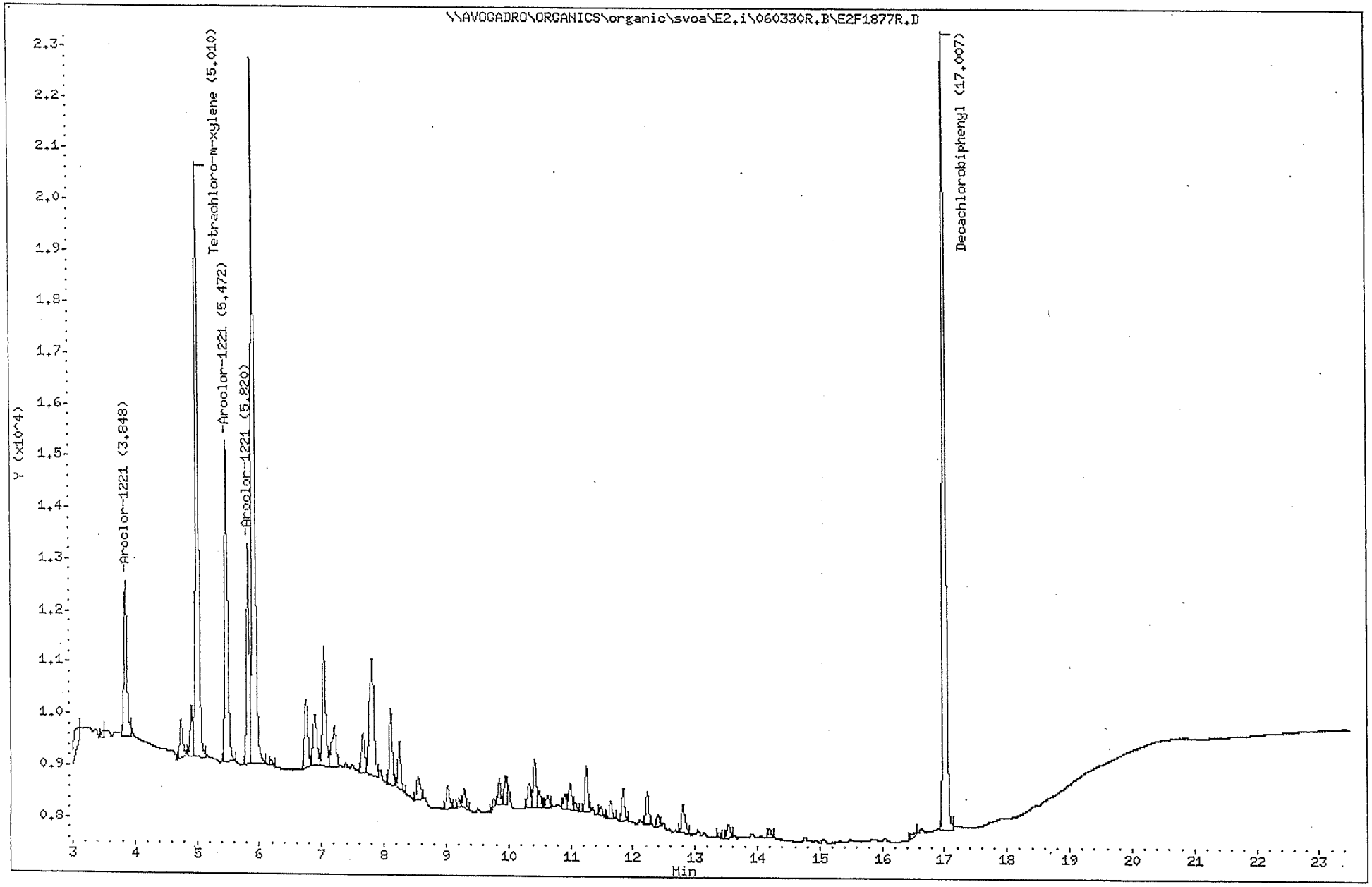
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1877R.D
Date : 30-MAR-2006 18:58
Client ID: AR1221FL3
Sample Info: AR1221FL3,AR1221FL3,,ar1221.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1877F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D
Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
Inj Date : 30-MAR-2006 18:58
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 1 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1221.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
6.41	6.41	0.000	13493	0.02000	0.020		(a)
2 Aroclor-1221					CAS #: 11104-28-2		
5.26	5.26	0.000	4734	1.00000	1.0	80.00- 120.00	100.00
7.20	7.20	0.000	7871	1.00000	1.0	146.27- 186.27	166.27
7.56	7.56	0.000	5525	1.00000	1.0	96.71- 136.71	116.71
Average of Peak Amounts =					1		
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
19.6	19.6	0.000	16954	0.02000	0.020		(a)

3/31/06

Data File: E2F1877F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1877R.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1877R.D
Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
Inj Date : 30-MAR-2006 18:58
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 1 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1221.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
5.01	5.01	0.000	11534	0.02000	0.020		(a)
2 Aroclor-1221					CAS #: 11104-28-2		
3.85	3.85	0.000	3031	1.00000	1.0	80.00- 120.00	100.00
5.47	5.47	0.000	6227	1.00000	1.0	185.44- 225.44	205.44
5.82	5.82	0.000	4291	1.00000	1.0	121.57- 161.57	141.57
Average of Peak Amounts =					1		
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
17.0	17.0	0.000	15520	0.02000	0.022		(a)

3/31/06 ✓

Data File: E2F1877R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

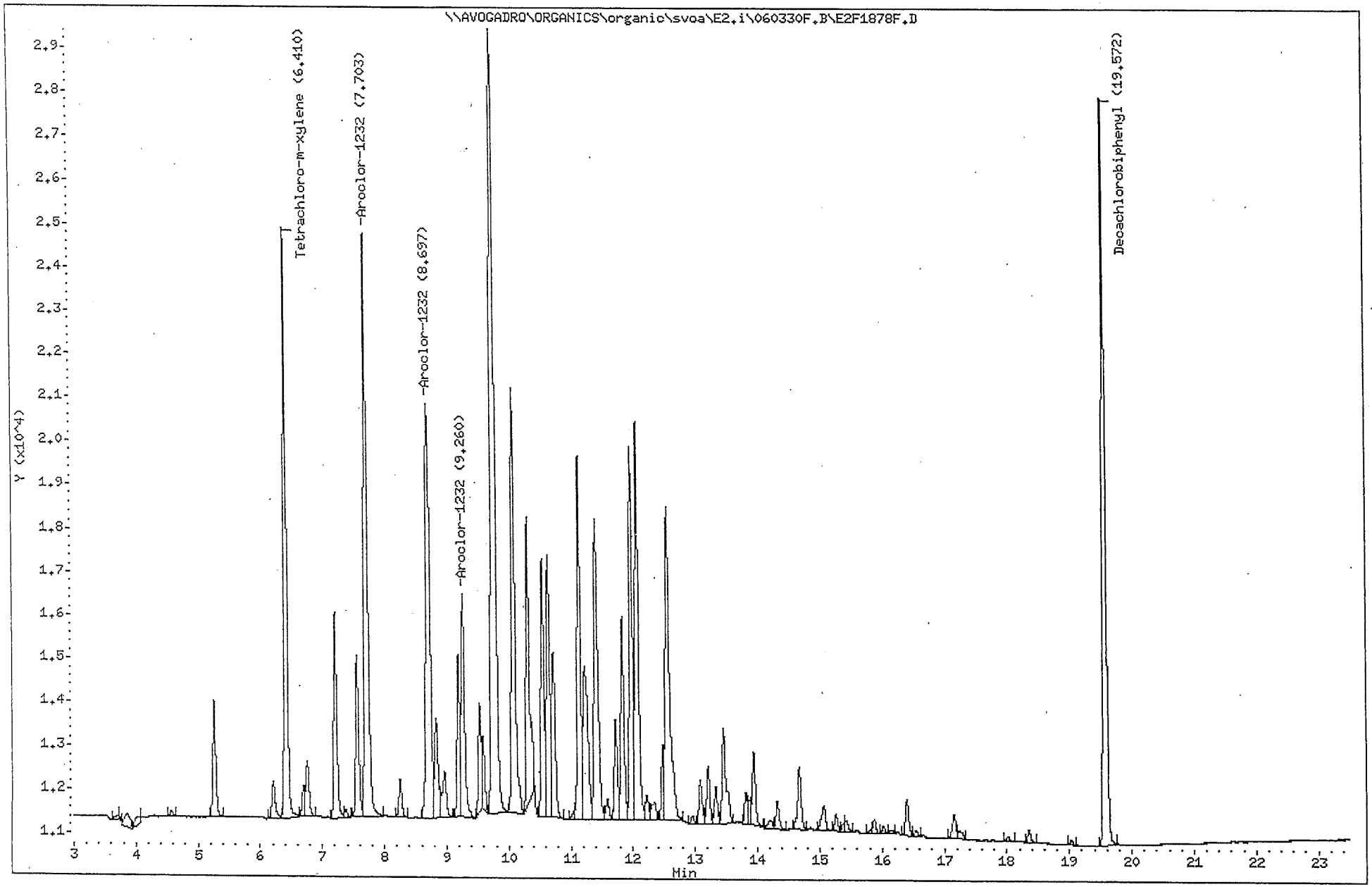
Figure 3

Aroclor 1232 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D
Date : 30-MAR-2006 19:24
Client ID: AR1232FL3
Sample Info: AR1232FL3,AR1232FL3,,ar1232.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

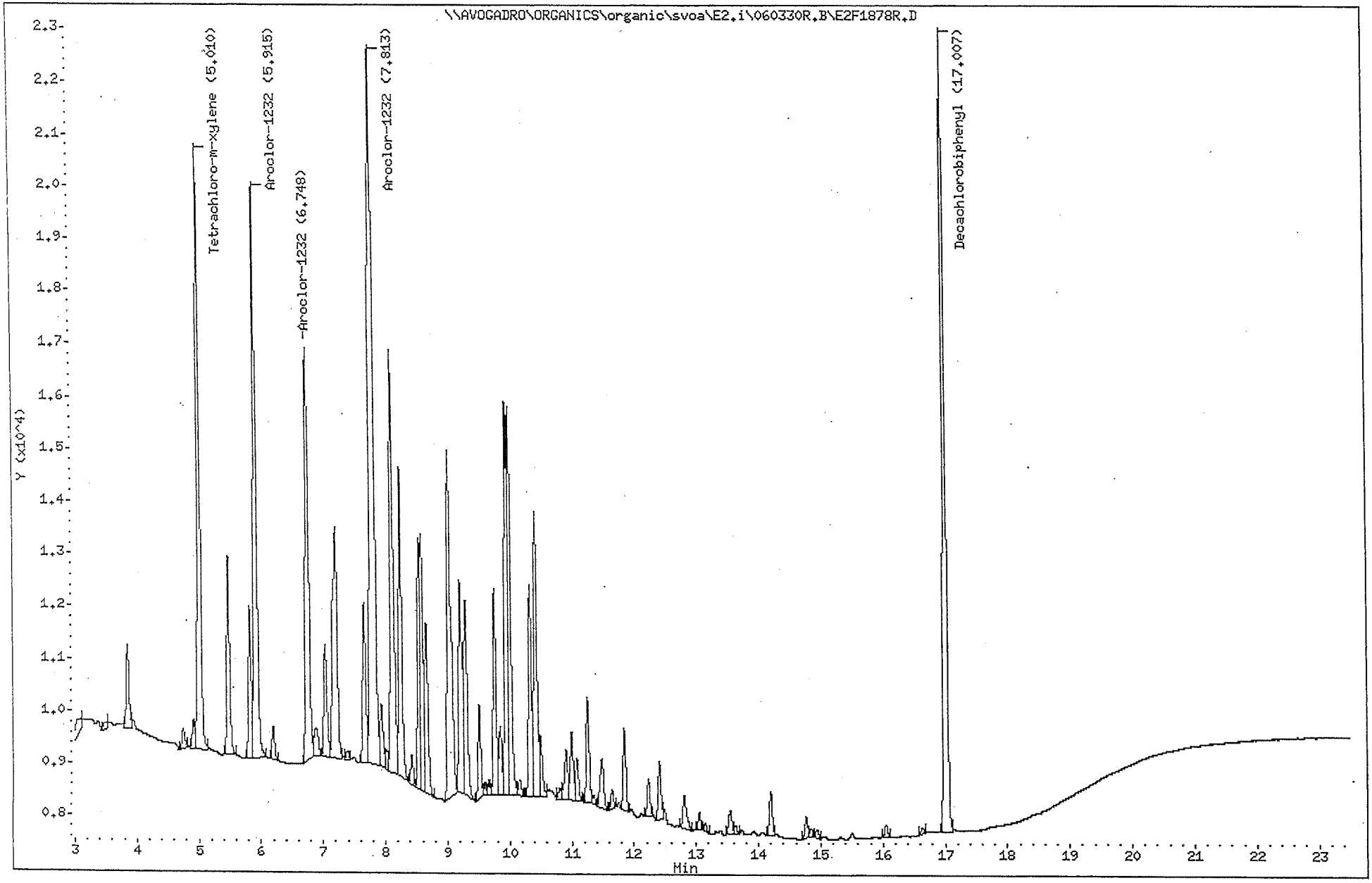
Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D
Date : 30-MAR-2006 19:24
Client ID: AR1232FL3
Sample Info: AR1232FL3,AR1232FL3,,ar1232.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1878F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D
Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
Inj Date : 30-MAR-2006 19:24
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 2 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1232.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	13570	0.02000	0.020			(a)
3 Aroclor-1232					CAS #: 11141-16-5			
7.70	7.70	0.000	13387	1.00000	1.0	80.00- 120.00	100.00	
8.70	8.70	0.000	9491	1.00000	1.0	50.90- 90.90	70.90	
9.26	9.26	0.000	5098	1.00000	1.0	18.08- 58.08	38.08	
Average of Peak Amounts =					1			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	17061	0.02000	0.020			(a)

3/31/06

Data File: E2F1878F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1878R.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D
Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
Inj Date : 30-MAR-2006 19:24
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 2 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1232.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	11550	0.02000	0.020			(a)
3 Aroclor-1232					CAS #: 11141-16-5			
5.92	5.92	0.000	10974	1.00000	1.0	80.00- 120.00	100.00	
6.75	6.75	0.000	7851	1.00000	1.0	51.54- 91.54	71.54	
7.81	7.81	0.000	13684	1.00000	1.0	104.69- 144.69	124.69	
Average of Peak Amounts =					1			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	15283	0.02000	0.022			(a)

3/31/06

Data File: E2F1878R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

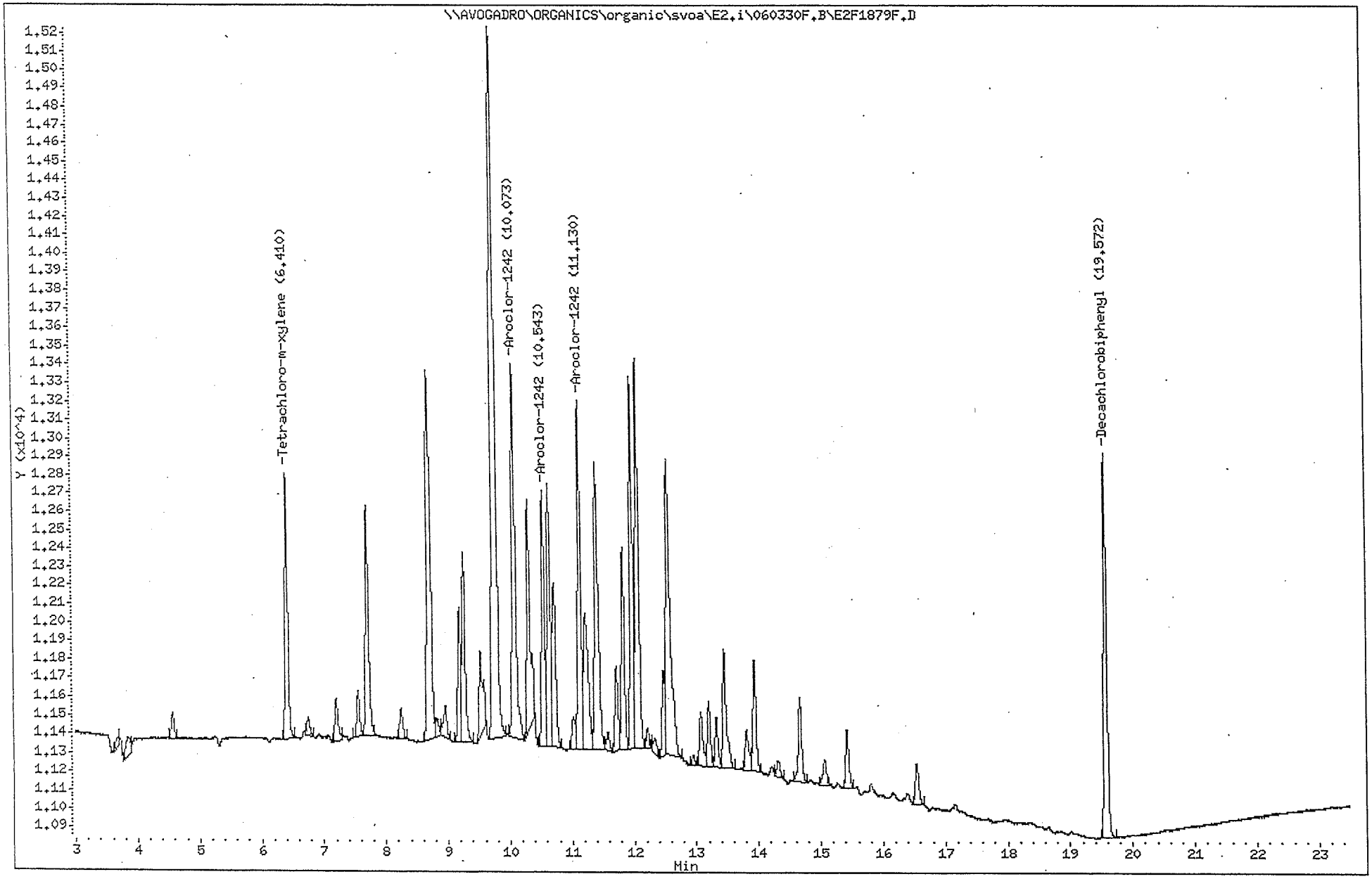
Figure 4

Aroclor 1242 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D
Date : 30-MAR-2006 19:51
Client ID: AR1242FL1
Sample Info: AR1242FL1,AR1242FL1,,ar1242.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

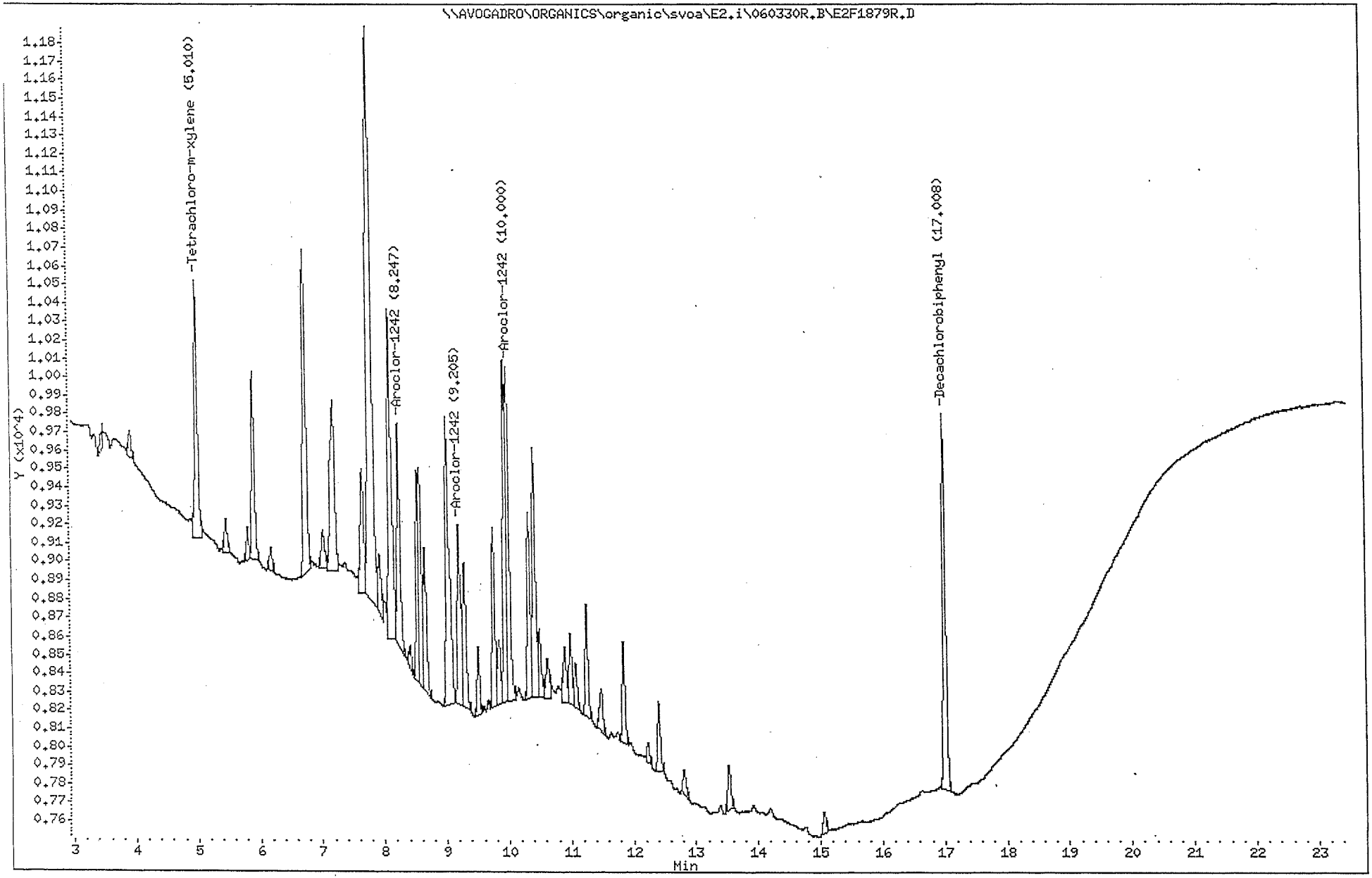
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D
Date : 30-MAR-2006 19:51
Client ID: AR1242FL1
Sample Info: AR1242FL1,AR1242FL1,,ar1242.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1879F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D
Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
Inj Date : 30-MAR-2006 19:51
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 3 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1242.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1442	0.00200	0.0021			(a)
5 Aroclor-1242					CAS #: 53469-21-9			
10.1	10.1	0.000	2024	0.10000	0.12	80.00- 120.00	100.00	(a)
10.5	10.5	0.000	1380	0.10000	0.12	44.06- 84.06	68.18	
11.1	11.1	0.000	1886	0.10000	0.12	71.10- 111.10	93.18	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2073	0.00200	0.0024			(a)

3/31/06

Data File: E2F1879F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1879R.D
Report Date: 31-Mar-2006 07:52

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D
Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
Inj Date : 30-MAR-2006 19:51
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:52 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 3 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1242.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1392	0.00200	0.0024			(a)
4 Aroclor-1242					CAS #: 53469-21-9			
8.25	8.24	0.010	1190	0.10000	0.12	80.00- 120.00	100.00	(a)
9.21	9.20	0.010	967	0.10000	0.12	62.62- 102.62	81.26	
10.0	10.0	0.000	1808	0.10000	0.13	115.66- 155.66	151.93	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2030	0.00200				(a)

3/31/06

Data File: E2F1879R.D
Report Date: 31-Mar-2006 07:52

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

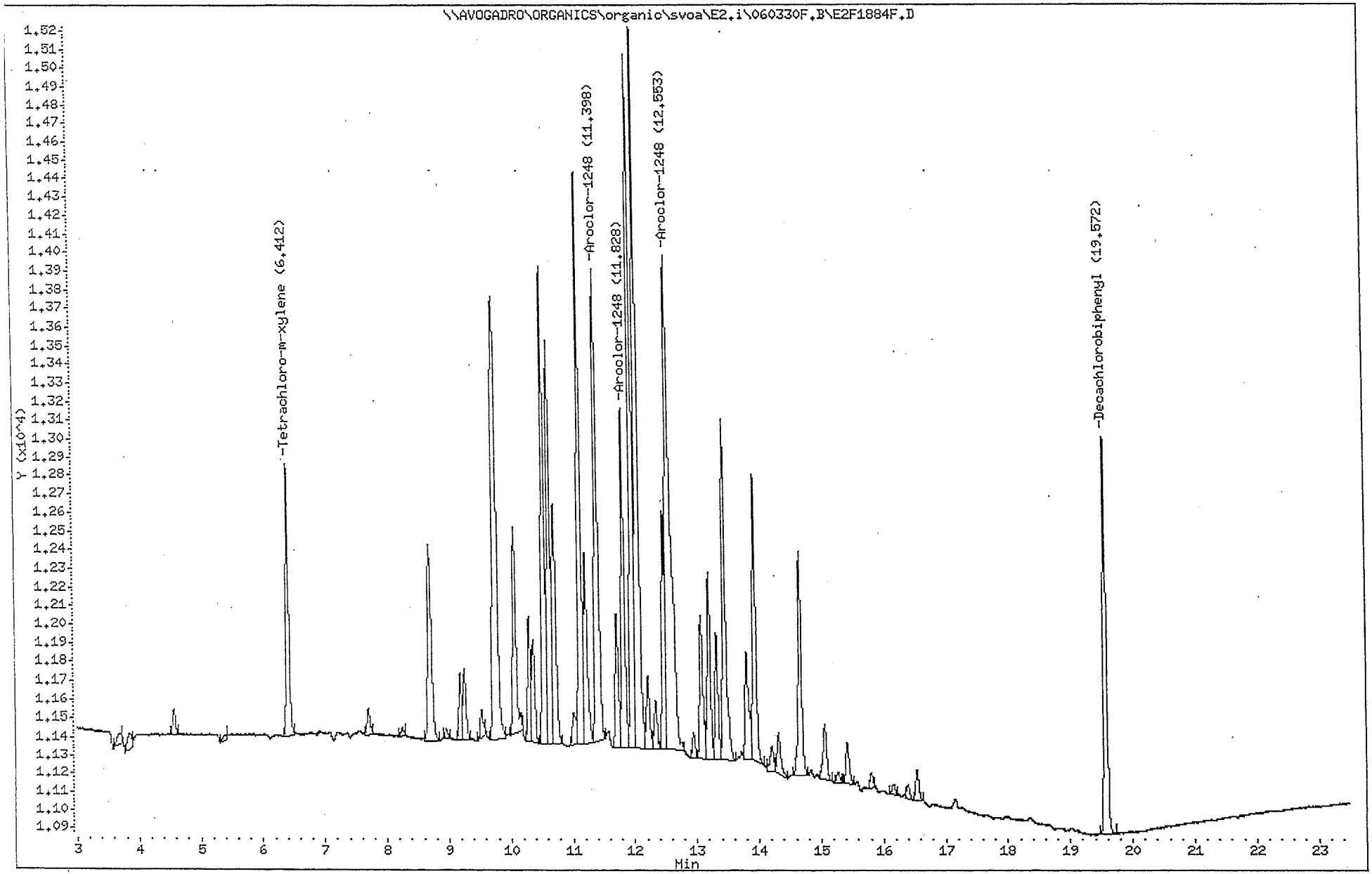
Figure 5

Aroclor 1248 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1884F.D
Date : 30-MAR-2006 22:04
Client ID: AR1248FL1
Sample Info: AR1248FL1,AR1248FL1,,ar1248.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

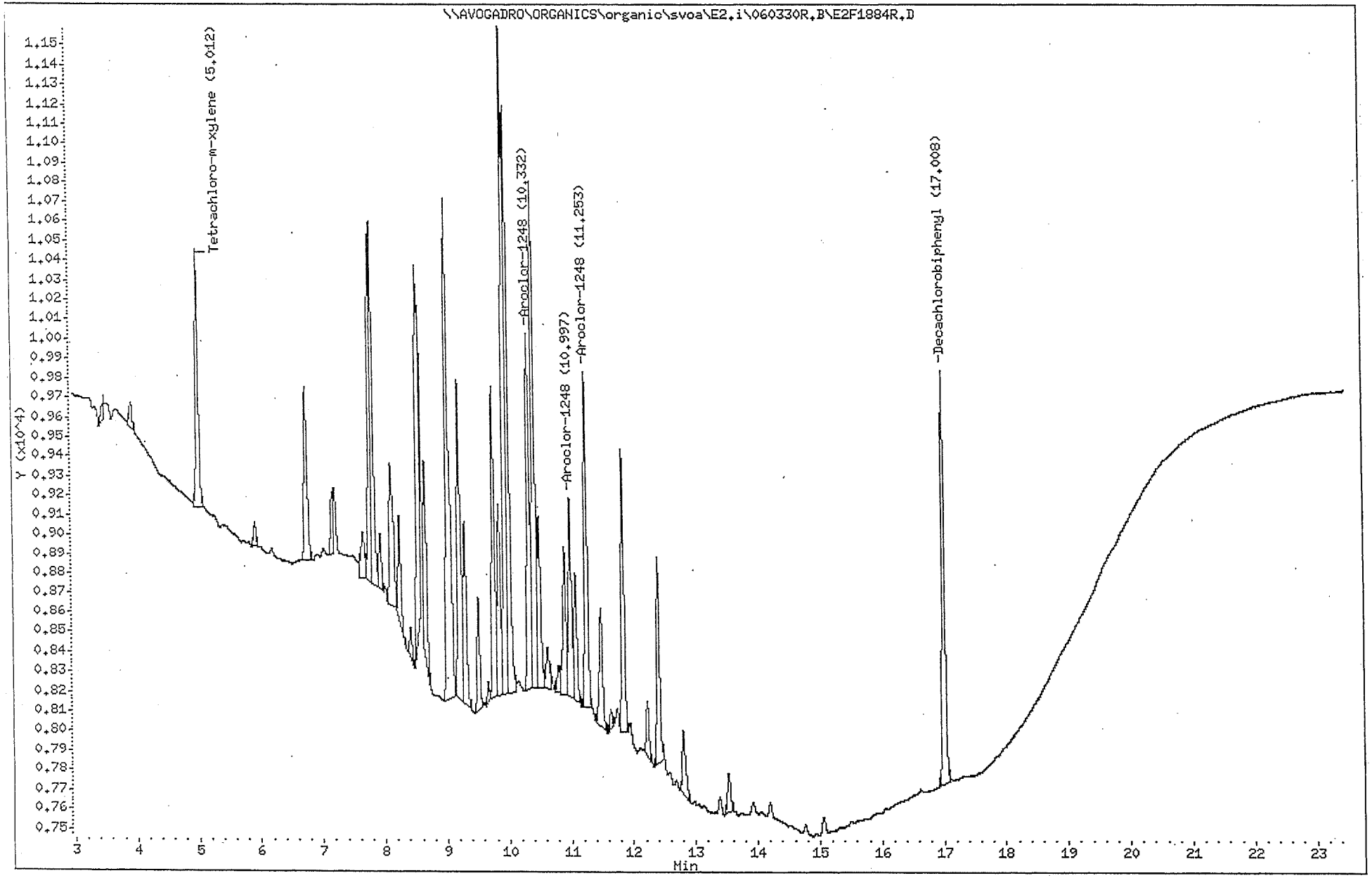
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D
Date : 30-MAR-2006 22:04
Client ID: AR1248FL1
Sample Info: AR1248FL1,AR1248FL1,,ar1248.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1884F.D
Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1884F.D
Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
Inj Date : 30-MAR-2006 22:04
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 8 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1248.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1454	0.00200	0.0022			(a)
6 Aroclor-1248					CAS #: 12672-29-6			
11.4	11.4	0.000	2529	0.10000	0.12	80.00- 120.00	100.00	(a)
11.8	11.8	0.000	1823	0.10000	0.12	50.48- 90.48	72.08	
12.6	12.6	0.000	2646	0.10000	0.12	89.75- 129.75	104.63	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2141	0.00200	0.0025			(a)

3/31/06

Data File: E2F1884F.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1884R.D
Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D
Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
Inj Date : 30-MAR-2006 22:04
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 8 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1248.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1315	0.00200	0.0023			(a)
5 Aroclor-1248					CAS #: 12672-29-6			
10.3	10.3	0.000	1811	0.10000	0.13	80.00- 120.00	100.00	(a)
11.0	11.0	0.000	1016	0.10000	0.12	37.21- 77.21	56.10	
11.3	11.3	0.000	1715	0.10000	0.13	72.29- 112.29	94.70	
Average of Peak Amounts =					0.13			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2112	0.00200				(a)

3/31/06

Data File: E2F1884R.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

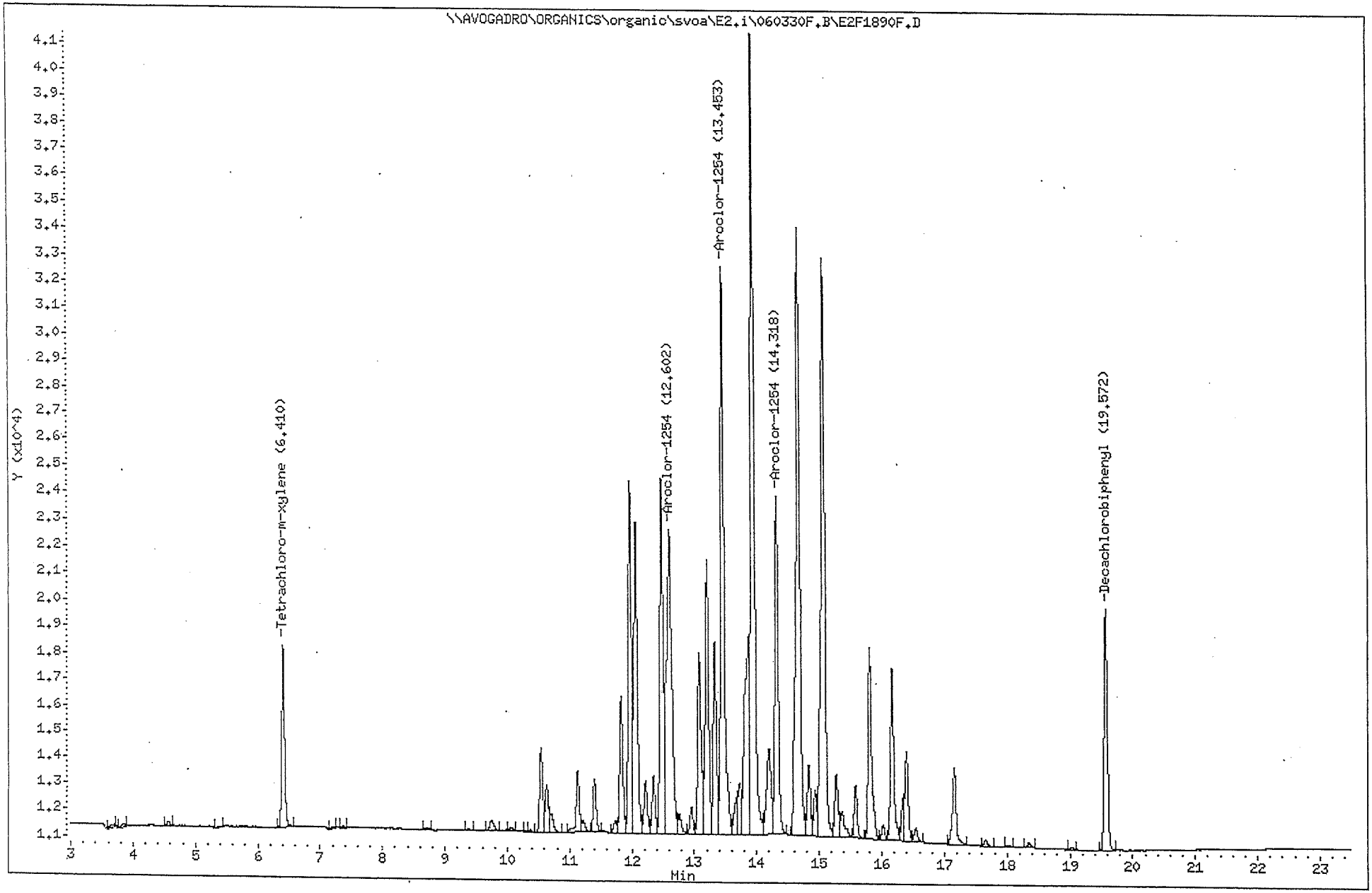
Figure 6

Aroclor 1254 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D
Date : 31-MAR-2006 00:44
Client ID: AR1254FL2
Sample Info: AR1254FL2,AR1254FL2,,ar1254.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

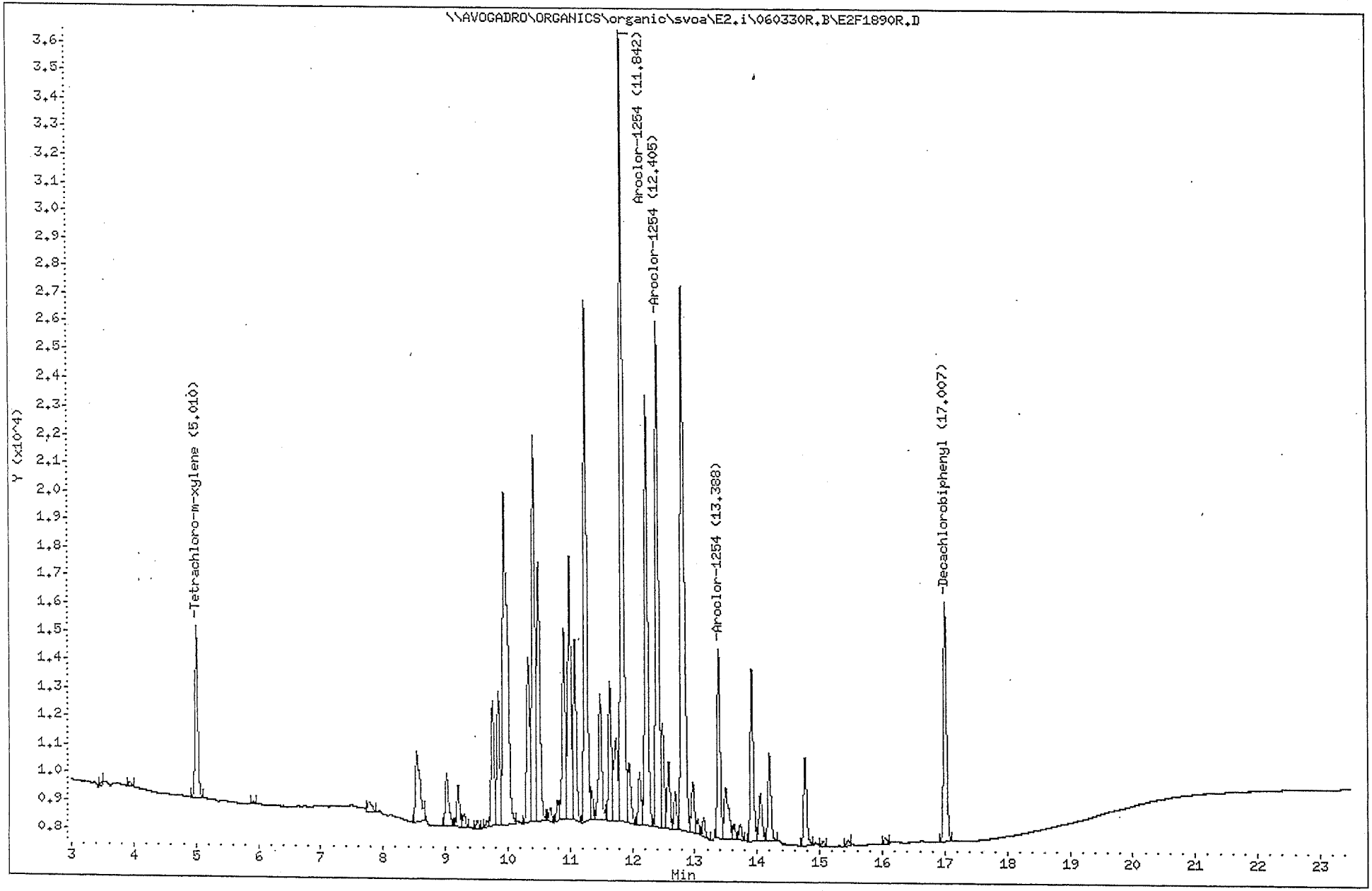
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1890R.D
Date : 31-MAR-2006 00:44
Client ID: AR1254FL2
Sample Info: AR1254FL2,AR1254FL2,,ar1254.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1890F.D
Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D
Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
Inj Date : 31-MAR-2006 00:44
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
Misc Info : 1,2,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 14 Calibration Sample, Level: 2
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1254.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
6.41	6.41	0.000	6923	0.01000	0.010		(a)
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
19.6	19.6	0.000	9157	0.01000	0.011		(a)
7 Aroclor-1254					CAS #: 11097-69-1		
12.6	12.6	0.000	11493	0.50000	0.54	80.00- 120.00	100.00(a)
13.5	13.5	0.000	21409	0.50000	0.54	171.37- 211.37	186.28
14.3	14.3	0.000	12764	0.50000	0.54	92.12- 132.12	111.06
Average of Peak Amounts =					0.54		

3/31/06

Data File: E2F1890F.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1890R.D
Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1890R.D
Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
Inj Date : 31-MAR-2006 00:44
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
Misc Info : 1,2,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 14 Calibration Sample, Level: 2
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1254.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	6063	0.01000	0.011			(a)
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	8543	0.01000	0.010			(a)
7 Aroclor-1254					CAS #: 11097-69-1			
11.8	11.8	0.000	28134	0.50000	0.50	80.00- 120.00	100.00	(a)
12.4	12.4	0.000	17965	0.50000	0.49	47.65- 87.65	63.86	
13.4	13.4	0.000	6744	0.50000	0.50	5.17- 45.17	23.97	
Average of Peak Amounts =					0.5			

3/31/06

Data File: E2F1890R.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Attachment 1

Corrective Action Examples and Documentation Tables

Attachment 1

Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria (RSD% >20% or r^2 < 0.990).	1. Check GC conditions such as temperature program, makeup gas flow rate, and carrier gas flow rate. Check if column bleeding occurs and more injection maintenance is needed. After the reasons are found, appropriate maintenance will be done and a new initial calibration will be run. If the new curve still fails, the reasons for failing the curve will be re-evaluated and another a new curve will be rerun after the reevaluation. If still not good, call GC manufacturer.	1. Note in instrument run logbook, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification (ICV) check does not meet QC criteria (D% > ±20%).	2. Check the preparation of ICV standard, and if necessary, make a fresh ICV standard. If it is found that ICV standard is ok, the standards for initial calibration will be checked such as evaporation and the preparation of standards. A new ICV and a new curve will be analyzed after problems are corrected.	2. Note in instrument run logbook, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria (D% > ±20%).	3. Maintenance such as replacing septum and liner for injection port will be done, and then a new set of CCVs and the samples related to the bad CCVs will be re-analyzed. If the CCVs still fail, A full maintenance including replacing septum, liner and gold seal and trimming the column will be done and a new initial calibration will be run. Then the CCV and/or QC and samples re-analyzed.	3. Note in instrument run logbook. If instrument maintenance performed, note in maintenance logbook.
4. Method blank contains target compound above reporting limit.	4. A new aliquot of the blank from prep lab will be reanalyzed to check if contaminants are carried over from previous dirty samples and/or from standards or a bad injection occurs. If the results from re-analysis of the	4. If the re-extraction/re-analysis meets the criteria, the results are reported. Document in the run log. If there is insufficient sample volume for re-extraction/re-analysis, report the initial results. Document in the run log,

	blank are still the same, whole batch of the samples that are related to the blank will be re-extracted and re-analyzed.	document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.
5. Surrogates in the method blank are outside of acceptable range.	5. A new aliquot of the blank from prep lab will be reanalyzed. If the results from re-analysis are the same, the samples that are related to this blank will be re-extracted and re-analyzed.	5. Same as documentation #5.
6. Compound out of acceptance range in laboratory control sample.	6. Investigate source of problem. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP/project requirements, this LCS will be reanalyzed. If the results of reanalysis are the same, the samples that are related to the LCS will be re-extracted and re-analyzed.	6. Flag all compounds out of range on Form 3 of data report, if samples are re-analyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook, comment in data review checklist to be included in project narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer. If re-analysis cannot be performed due to insufficient sample, comment in data review checklist to be included in project narrative, and document as above.
7. Surrogates in samples are out of control limits.	7. 7.1 No actions are needed for the following situations: • One of the surrogates (TCX and DCB) is in control limits, and the other is above the upper limit due to coelution with contaminants. • TCX is in the control limits, and DCB is below the lower limit. This is possibly due to matrix effect. 7.2 Further actions are needed for the following cases: • Both of the surrogates (TCX and DCB) do not meet criteria. • TCX is lower than the lower limits and DCB is	7. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, comment on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. Document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer.

	in the limits. This is most possibly due to evaporation of TCX in the extracts. Actions for 7.2: The samples will be reanalyzed. If the results of re-analysis are the same, the samples will be re-extracted and re-analyzed.	
8. Compound in sample exceeds upper calibration standard concentration.	8. Re-analyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running an instrument using auto-sampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be re-analyzed if sufficient volume exists.	8. Note in instrument run log, comment on data review checklist to be included in project narrative.
9. Instrument blank (GC) contains contamination above QC criteria.	9. Investigate source of problem, decontaminate purge and trap instrument, re-analyze blank and all effected samples.	9. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, comment on data review checklist to be included in project narrative.

10. Matrix spike recovery out of QC range.	10.Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in un-spiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, re-analyze spike sample.	10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
11. Duplicate (or MSD) relative percent difference exceeds QC limit.	11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, re-analyze duplicate sample.	11. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
12. Retention Time shift.	12. A. No further action is required if the retention times of the CCV and samples shift after regular maintenance such as replacing the septum, liner, gold seal and trimming of the columns. The retention times will be established. B. If the retention times of the CCV shift during analysis, GC maintenance will be performed and the CCV and associated samples and QC will be re-analyzed. C. If the retention times of the CCV do not shift, but the retention times of surrogates in some of the samples do shift, the samples will be re-analyzed. If the retention times still do not meet the criteria, matrix affect is assumed and the results will be reported. This situation will be documented.	12. Document in the run log. If CCV, QC, and samples were re-analyzed, document in the run log. If the retention times shift with respect to re-analysis, document in the run log, in the checklist for the case narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date.

Attachment 2
Department of Defense QC Requirements

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	NA	NA	
Breakdown check (Endrin/ DDT Method 8081 only)	Daily prior to analysis of samples	Degradation ≤ 15% for both Endrin and DDT.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15%.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below (except for Method 8082, which may only use Option 1 or 2): Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (in Method 8081A exclude toxaphene and technical chlordane, in Method 8015B exclude GRO, DRO, and residual range organics (RRO)).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

TABLE D-15. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 SOLID MATRIX[26]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
4,4'-DDD	81	18	30	135	10	155
4,4'-DDE	97	10	70	125	60	135
4,4'-DDT	92	16	45	140	30	155
Aldrin	93	16	45	140	30	155
alpha-BHC	93	10	60	125	50	135
alpha-Chlordane	92	10	65	120	55	130
beta-BHC	95	11	60	125	50	135
delta-BHC	94	12	55	130	45	145
Dieldrin	96	10	65	125	55	135
Endosulfan I	74	20	15	135	10	155
Endosulfan II	89	17	35	140	20	160
Endosulfan sulfate	99	12	60	135	50	145
Endrin	97	12	60	135	50	145
Endrin aldehyde	92	18	35	145	20	165
Endrin ketone	100	11	65	135	55	145
gamma-BHC	91	11	60	125	50	135
gamma-Chlordane	96	10	65	125	55	135
Heptachlor	96	15	50	140	35	155
Heptachlor epoxide	98	11	65	130	55	140
Methoxychlor	100	14	55	145	45	155

TABLE D-16. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 WATER MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	85	20	25	145
Aroclor 1260	87	19	30	145

TABLE D-17. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 SOLID MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	90	16	40	140
Aroclor 1260	96	12	60	130

[26] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

[27] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Preparation of Aqueous Samples by Acid Digestion for ICP

SOP No. 100.0003

Rev. 7

	Signature	Date
QA Director:		3/16/09
Lab Director:		3/16/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Sample Preparation of Aqueous Samples by Acid Digestion for ICP

Rev. 7

1. Scope and Applications

This Standard Operating Procedure (SOP) deals with the preparation of aqueous samples for analysis by USEPA SW846 methods 6010 and 6020A. Discussion includes sample extraction and sample concentration technique and analysis of dissolved metals or total/ total recoverable metals in aqueous samples using methods SW-846 SW3005/3010.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date, and that it is implemented appropriately. Supervisors/lab managers review the logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 50mL aliquot of an aqueous sample is digested with acid prior to metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been modified from the original 100mL sample volume to reduce reagent use and acid waste. All reagents have been adjusted accordingly. ICP-MS preparation has been modified to utilize a lower volume of HCL to minimize molecular-ion interferences. Refer to Chapter 2 of SW-846 for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The hold time for ICAP analysis is 180 days from the date of sample collection.

4.2 Aqueous samples for Total recoverable metals analysis must be preserved with concentrated HNO_3 to a pH of less than 2 and stored in an amber glass or polyethylene bottle.

4.3 Aqueous samples for dissolved metals must be filtered through 0.45um filter paper prior to preservation with concentrated HNO_3 to a pH <2.

5. Interferences and Potential Problems

Possible contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware contaminated.

5.3 Acid dispensers.

5.4 DI water rinsing bottles contaminated.

5.5 Poor lab technique.

5.6 Cross-contamination from high-level samples.

5.7 Sample matrix effects: Extreme organic samples.

6. Equipment and Apparatus

Equipment used in this preparation method include:

6.1 10% HNO_3 acid bath, reagent grade acid.

6.2 50 mL Polyethylene centrifuge/digestion tubes with caps.

6.3 Polyethylene ribbed Watch Glasses.

6.4 Whatman 41 filter paper (if necessary).

6.5 50 ml Plastic funnels (if necessary).

6.6 Thermometers- NIST Calibrated (0-100°C)

6.7 Hot plates with graphite tube holders for even heating.

6.8 Wide range pH paper, 0-14, Whatman ColorPHast.

6.9 Narrow range pH paper 0-2.5, Whatman ColorPHast.

6.10 Eppendorf and/or Wheaton pipettes.

6.11 Beckman GPR Centrifuge.

7. Reagents

The following reagents are used during sample preparation:

7.1 Concentrated HNO_3, Fisher, ACS trace metals grade.

7.1.1 1:1 (v/v) HNO_3 Fisher, ACS trace metals grade.

7.2 Concentrated HCl, Fisher, ACS trace metals grade.

7.2.1 1:1 (v/v) HCl, Fisher, ACS trace metals grade.

7.3 Spiking solutions, High Purity Standards.

7.4 Laboratory Control Sample Solutions, High Purity Standards.

8. Procedure

8.1. Digestion of highly colored total water samples for ICAP Analysis **(Method 3010)**:

Enter Lab ID #'s of all the samples to be digested into the proper column in the Sample Digestion Logbook. All volumes of reagents added as well as the initial and final volumes must be entered in this Logbook.

8.1.1. The sample pH should have been checked at the time of reciept in sample Log-in. Prior to analysis, re-check the pH for aqueous samples using the wide range pH paper (pH should be <2). If the pH strip color is more red than magenta, or there is any doubt of the pH being less than 2, validate the pH with the narrow range pH paper which measures pH 0 to 2.5. Enter observations in the appropriate column in the Logbook and adjust and note the pH if not less than 2. If pH adjustment needs to be done, be sure to notify the client first. Acidify the sample with concentrated HNO_3 to a pH <2. Document all pH adjustments. Shake sample well and let it sit for 24 hours before digestion.

8.1.2. Transfer 50 mL of sample into a plastic digestion tube using the graduated marking on the tube to reach volume.

8.1.3. Add 1.5mL conc. HNO_3 to the sample.

8.1.4. Cover with a ribbed watch glass.

8.1.5. Place the tubes in the graphite holders, on a hotplate, inside the inorganic hood (PVC ducting).

8.1.6. Heat at 95°C (90°C – 95°C is an acceptable temperature range) until the sample volume has been reduced to approx. 2.5 –5.0mL. Do not boil the sample or let it go dry.

8.1.7. Take tubes off the hotplate and allow them to cool to room temperature.

8.1.8. Add another 1.5mL of conc. HNO_3 to each sample.

8.1.9. Heat the sample again and reduce to 1.5 –3.0 mL, ensuring a reflux action occurs.

8.1.10. Continue to add HNO_3 in 1.5mL aliquots until digestate appears pale in color. (Add no more than 5mL conc. HNO_3.)

8.1.11. Cool the tube and add 5mL of 1:1 (v/v) HCl.

8.1.12. Heat for an additional 15 minutes.

8.1.13. Rinse the walls of tube with DI water.

8.1.13.1. If the sample is not to be filtered, volumize the sample back to 50 ml with DI water using the graduation on the tube to obtain the proper volume.

8.1.13.2. If sample needs to be filtered, volumize the sample to 50 ml and filter through Whatman 41 filter paper. Do not rinse filter.

8.1.14. Assign the samples to a preparation batch using Mitkem's LIMS system.

8.1.15. Transfer logbook and digestates to the Metals Instrument Lab.

8.2. Digestion of Dissolved or Total/Total Recoverable Aqueous Samples and Leachates for ICAP Analysis **(Method 3005)**:

Enter Lab ID# of all samples to be digested into the proper column in the Sample Digestion Logbook. All volumes of reagents added as well as the initial and final volumes must be entered in the Logbook.

8.2.1 Dissolved samples must be filtered first through 0.45um filter paper and preserved with conc. HNO_3. Typically this is done in the field however, if samples arrive at Mitkem requiring filtration, it should be done ASAP. Our general goal is within 24 hours of receipt.

8.2.2 Transfer a 50mL aliquot of the sample to a plastic digestion tube using the graduated marking on the tube to reach volume.

8.2.2.1 Add 1.0mL of conc. HNO_3 and 2.5mL of conc. HCL for ICP-AES samples (SW6010).

8.2.2.2 Add 1.0mL 1:1 HNO_3 and 0.5mL of 1:1 HCL for ICP-MS samples (SW6020).

8.2.3 Heat to 90-95°C in graphite holders on hotplates in the hood. Cover each tube with a ribbed watch glass and evaporate until the sample volume equals 7.5-10 mL.

8.2.4 Remove tube and allow to cool.

8.2.5 Rinse the walls of the beaker with DI water and volumize to 50mL with DI water.

8.2.6 If insoluble material remains, volumize sample to 50 mL and filter through Whatman 41 filter paper, or centrifuge.

8.2.7 ICP-MS samples may be recorded in the Method E200.7 E200.8 Aqueous Metal Prep Logbook (**figure 2**) as the concentration of the acids are the same. Make sure to circle the method SW3005. Initial and date all crossouts.

8.2.8 Record all digestion information in the Aqueous Metal Prep Logbook (**figure 1**).

8.2.9 Assign the samples to a preparation batch using Mitkem's LIMS system.

8.2.10 Transfer logbook and digestates to the Metals Instrument Lab.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic Prep Lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1. Method blanks:

A method blank must be run with every discrete batch of samples that is prepped. The number of samples per batch may not exceed 20.

10.1.1 Use 50mL of DI water as the method blank for aqueous sample preparation. Prep this sample as described in **Section 8**. Label appropriately.

10.2. Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch may not exceed 20.

10.2.1 For ICP-AES samples: measure approximately 40 ml of DI water as if it were a sample and add High Purity Standard CLP-CV-1 at 455µL; CLP-CV-2 and CLP-CV-3 at 45.5 µL each. Prep this sample as described in **Section 8**. Label appropriately.

10.2.2 For ICP-MS samples: measure approximately 40 ml of DI water as if it were a sample and add High Purity Standard CLP-CV-1 at 200µL; CLP-CV-2 and CLP-CV-3 at 20 µL each. Prep this sample as described in **Section 8**. Label appropriately.

10.3. Matrix Spikes and Duplicate Samples:

10.3.1 With at least every 20 samples a sample duplicate and sample spike (containing all target analytes)must be prepped and analyzed by the laboratory. Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 50mL of sample (the same sample).

10.3.1.1 Spike Sample:

- For ICP-AES samples: add three spiking standards; High Purity Standard CLP-CV-1 at 455µL; CLP-CV-2 and CLP-CV-3 at 45.5µL each to the sample. Label this sample: "LabID"'MS'.
- For ICP-MS samples: add three spiking standards; High Purity Standard CLP-CV-1 at 200µL; CLP-CV-2 and CLP-CV-3 at 20µL each to the sample. Label this sample: "LabID"'MS'.

10.3.1.2 Sample Duplicate:

Label the other additional 50mL of sample as "LabID"'D'. This is an exact duplicate of the original sample. Digest the sample duplicate as described in **Section 8**.

10.4 Standard Preparation

10.4.1 All standards made from a primary standard expire on or before the primary standard's expiration date. For standards without manufacturer-designated expiration dates, the expiration date will be 1 year from receipt.

10.5 Digestion Tubes

10.5.1 All lots of digestion tubes must be tested for conformance. A group of 10 tubes are measured for volume by weight. The weights are recorded and the average weight is calculated. If the average weight is less than or equal to 3 % of the true value, the lot of tubes are considered valid.

10.6 Pipettes: Two sizes of pipettes are used in the method.

10.6.1 0 to 1000 µL: Five replicate weights are recorded. The average of the five replicates is calculated. If the average weight is less than or equal to 3 % of the true value, the pipette is acceptable for use.

10.6.2 0 to 100 µL: Five replicate weights are recorded. The average of the five replicates is calculated. If the average weight is less than or equal to 1 % of the true value, the pipette is acceptable for use.

11. Data Validation and Reporting

Data generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the manager.

12. Corrective Action Procedures

12.1 If the method blank concentration for any analyte is greater than the established method criteria, the samples will be scheduled for re-prep by the metals analyst/supervisor.

12.2 If the LCS is outside of control limits, the samples will be scheduled for re-prep by the metals analyst/supervisor for those elements that failed.

12.3 If a Spike sample was not spiked, the sample will be scheduled for re-prep by the metals analyst/supervisor.

13. Health and Saftey

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. Reference

1. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Update 3 Method 3005.

2. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Update 3 Method 3010.

3. U.S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Chapter Three: Inorganic Analytes. Revision 4, February 2007.

Attachments

Figure 1: Aqueous Metal Prep Logbook
Figure 2: Method E200.7 Aqueous Metal Prep Logbook

Figure 1
Aqueous Metal Prep Logbook

MITKEM LABORATORIES: Aqueous Metals Preparation Logbook

Date	Sample ID		Client ID	Sample Vol (ml)	pH	Sample Color Before	Sample Clarity Before	Conc. HNO_3 (ml)	Conc. HCl (ml)	1:1 HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments	Analyst

HCI Lot# _____

HNO3 Lot# _____ **Method:** _____ **Digestion Temp:** _____ **oC**

LCSS/Spike ID: _____

_____

_____ **SOP#:** _____

Digestate Relinquished to: _____

Logbook ID 100.0125 -12/08

REVIEWED BY: _____

Figure 2

Method E200.7 Aqueous Metal Prep Logbook

MITKEM LABORATORIES: Aqueous Metals Preparation Logbook

Date	Sample ID		Sample Vol (ml)	Sample pH	Sample Color Before	Sample Clarity Before	1:1 HNO_3 (ml)	1:1 HCI (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments	Analyst

HCI Lot# _____

HNO3 Lot# _____

Method (circle) : **E200.7** **200.8** **SW-3005 ICP/MS**

Digestion Temp: _____ **oC**

LCSS/Spike ID: _____

_____

_____

Digestate Relinquished to: _____

Logbook ID 100.0130 -05/09

REVIEWED BY: _____

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURES

for

Mercury Analysis in Aqueous and Soil Samples by Flow Injection Mercury System (FIMS) for Atomic Absorption

by

SW846 Method 7470A/7471B

Rev. 9

	Signature	Date
QA Director:		11/6/08
Lab Director:		11/6/08

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Mercury Analysis in Aqueous and Soil Samples
by Flow Injection Analysis System
for Atomic Analysis

by

SW846 Method 7470Aand 7471B

Rev. 9

1. Scope and Application

This SOP describes the procedures applicable to the preparation and analysis of mercury in aqueous and soil samples. Matrices include ground waters, aqueous samples, soils, sediments, sludges, and TCLP. All samples require digestion prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 The aqueous samples are digested with concentrated HNO_3, concentrated H_2SO_4, potassium permanganate and potassium persulfate at 95°C. The procedure converts various organically bound compounds and inorganic forms

of mercury into mercuric ions, which can be analyzed with a Flow Injection Analysis System (FIAS) for atomic spectroscopy. The soil/sediment samples are digested using aqua regia and potassium permanganate at 95±3°C and analyzed same as the aqueous samples.

3.2 The mercury ions formed during the digestion step are reduced to the elemental state and aerated into an absorption cell. Absorbance is measured at 253.7nm and is a function of mercury concentration.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For mercury analysis by SW 846 method 7470A water samples are collected in one-liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 All samples are stored at room temperature until analyzed.

4.3 Sample hold time for mercury analysis by SW 846 methods 7470A and 7471B is 28 days from date of sample collection.

5. Interferences and Potential Problems

5.1 Sulfides at levels above 20 mg/L or 20mg/Kg interfere. Potassium permanganate is added to the samples to eliminate possible interference of sulfides.

5.2 Copper at levels above 10 mg/L or 10mg/Kg interfere.

5.3 Seawaters, brines and industrial effluents high in chlorides interfere and require additional potassium permanganate for conversion to free chlorine. Free chlorine also absorbs radiation at 253.7nm. Therefore, the free chlorine is removed by addition of hydroxylamine sulfate reagent. In addition, the dead air space in the BOD bottle must be purged before adding the stannous sulfate.

5.4 Some volatile organic materials absorb at 253.7nm and may interfere.

6. Equipment and Apparatus

Equipment and instrumentation used in this analysis method include:

6.1 Equipment:

6.1.1 Perkin-Elmer FIMS 100.

6.1.2 Printer.

6.1.3 Wheaton BOD bottles.

6.1.4 Top loading balance - capable of accurate measurement to 0.01gram.

6.1.5 Hot Plate with graphite block digestor

6.2 Preventative Maintenance:

6.2.1 Pump tubing is replaced every 48 hours of instrument run time.

6.2.2 The windows of the optical cell are cleaned whenever the cell is replaced.

6.2.3 The inside of the optical cell is cleaned once every 48 hours of instrument run time.

6.3 Troubleshooting - Refer to the FIMS Analysis manual.

6.4 Glassware

6.4.1 100mL Class "A" volumetric flasks.

6.4.2 Class "A" volumetric pipettes ranging from 10µL to 1.0mL.

6.4.3 100 and 250mL Class "A" graduated cylinder or equivalent.

7. Reagents

7.1 Sulfuric Acid: Ultra Trace Grade

7.2 Nitric Acid: Ultra Trace Grade

7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Stannous chloride solution:
15 g $SnCl_2$ to 1000mLs of 3% HCl solution.

7.5 Sodium chloride-hydroxylamine sulfate solution:
12 g NaCl and 12g hydroxylamine sulfate to 100mLs of reagent water.

7.6 5% Potassium permanganate solution:
Dissolve 50g of $KMnO_4$ in 1000mLs of DI H_2O.

7.7 5% Potassium persulfate solution:
Dissolve 50g of $K_2S_2O_8$ in 1000mL of DI H_2O.

7.8 Stock mercury solution(Primary):
1000 mg/L purchased commercially.

7.9 Stock mercury solution(Independent Source):
1000 mg/L purchased commercially.

7.10 Mercury standards are stored in Wet Chem cupboards. Expiration dates for the standards are either the date one year from date of receipt or that designated by the manufacturer whichever comes first. Reagents and intermediate standards have a 28-day hold time.

7.11 3% HCl: 30mL concentrated HCl diluted to 1L with DI H_2O.

7.12 HCl: conc., Ultra Trace grade.

7.13 Aqua Regia: prepare immediately before use by carefully adding three volumes of conc. HCl to one volume HNO_3.

8. Procedure

8.1 Preparation

8.1.1 Aqueous Samples: Transfer 100mL of sample or an aliquot diluted to 100mL to a reagent water-rinsed BOD bottle with a graduated cylinder. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool samples and add 6mL sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved.

Record all volumes and reagents in the Mercury Digestion logbook , **Attachment 1**. Immediately prepare for analysis.

8.1.2 Soil Samples: Weigh a 0.50 – 0.60g portion of a well homogenized untreated sample and place in the bottom of a BOD bottle. See SOP 110.0039 for sub-sampling techniques. Add 5mL of reagent water and 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50mL reagent water and 15mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool and add 6mL of the sodium chloride – hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved. Add reagent water to a final volume of 100mL. Pour an aliquot into a polyethylene tube for analysis. Be careful to avoid pouring the sediment into the tube. Record all volumes and reagents in the Mercury Digestion logbook, **Attachment 1**.

8.2. Calibration Standards

8.2.1 Working Calibration standards are prepared from a mercury intermediate standard at 500µg/L. The intermediate is prepared by pipetting 50µL of 1000mg/L Primary stock standard into a 100mL volumetric flask. Bring up to volume with 3% HCl.

Label standard as IIyymmdd#, where:

I I = Inorganic Intermediate
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

All standard preparation information is documented in the Metals Standard Receipt/Preparation Logbooks (Primary, Intermediate and Working).

Into BOD bottles containing approximately 10mL DI H_2O, pipet the following volumes of intermediate standard in order to achieve the corresponding final working standard concentrations:

Volume	Concentration (µg/L Hg)	Standard ID
40µL	0.2	S1
200µL	1.0	S2
400µL	2.0	S3

1mL	5.0	S4
2mL	10.0	S5
0mL	0.0	S0

8.2.2 Depending on the matrix of samples being prepared, follow the digestion as follows:

8.2.2.1 Soil Calibration standards, add 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50 mL reagent water and 15 mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved. Calibration working standards are prepped with each batch of samples and undergo a digestion of at least 30 minutes. Soil standards are brought up to 100mL final volume with DI H_2O prior to analysis.

8.2.2.2 Aqueous Calibration standards, add approximately 90 mL DI H_2O to each BOD bottle. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. Do not volumize to 100 mL with DI H_2O prior to analysis. **CAUTION**: do this addition under the hood, Cl_2 could be evolved

8.2.3 A Calibration Blank, ICB/CCB, is prepared as the S0 calibration standard.

Sample concentrations are not reported below the lowest calibration standard of 0.2ug/L (aqueous samples, or 0.033 mg/Kg for soils) without method modification. This effectively becomes the method reporting limit (MRL) also referred to as Project Quantitation Limits (PQL).

8.2.4 ICV/CCV Intermediate standards are prepared at 5.0µg/L from an **Independent Source** Hg Primary stock standard. The intermediate

Hg CCV standard is prepared by pipetting 50µL of 1000mg/L Hg into a 100mL volumetric flask. The standard is brought to volume with 3%HCl for a final concentration of 500ug/L.

8.2.5 The working ICV/CCV is prepared by pipetting 1mL of the above intermediate standard into a BOD bottle and digesting as in **section 8.2.2.** An ICV/CCV is prepped with each batch of samples. Final concentration is 5µg/L and the standard is labeled as a mercury working calibration standard:

Label standard as IWyymmdd#, where:

I W = Inorganic Working
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

8.2.6 LCS/Matrix spike standard is also an intermediate concentration standard that is prepared by combining 45.5 uL of the **Independent Source** Hg Primary stock standard into a 100 mL volumetric flask and volumized to 100 mL with 3% HCl. The standard is labeled as for Hg working standards, as in **section 8.3**.

8.2.7 1mL of the above LCS/Matrix spike is added into 100 mL of DI H_2O and digested as an aqueous sample for the LCS-Water (LSCW) as in **section 8.1.1** . The true value of the LCS = 4.55µg/L at the instrument level.

8.2.8 1mL of the LCS/spike working spike is added to 0.6 g of acid-washed Teflon chips used to simulate a soil matrix for the LCS-Soil (LCSS). The true value of the LCS = 4.55µg/L at the instrument level.

8.2.9 1mL of the LCS/spike working spike is added to a chosen sample, and digested as per the sample matrix. This is the Matrix Spike. At the time of analysis, the spike = 4.55µg/L at the instrument level, times any sample dilution factor.

8.3 Instrumental Analysis

8.3.1 Adjust the argon pressure to 44psi for FIMS analysis. Turn on the FIMS system with the auto-sampler. Turn on the computer, printer. Allow the lamp to stabilize for 35-45 minutes.

8.3.2 Prepare the reductant solution, stannous chloride in **section 7.4**, and the carrier solution, 3% HCl in **section 7.11**.

8.3.3 Bring up the appropriate element file (Hg comm).

8.3.4 Set up the sample info file to coincide with the locations and sample identifications that will be analyzed in the run.

8.3.5 Fill out the Automated Control Window:

8.3.5.1 Type in a data file name.

8.3.5.2 Type in the name of the sample info file.

8.3.6 Load the auto-sampler tray.

8.3.7 Place the carrier tubing inlet into the carrier solution, and the reductant tubing inlet in the reductant solution. The reductant solution tubing has a red tab on it, and the carrier solution a yellow tab. Remove the cap from the gas chamber and start the pump to ensure it is working properly. Turn off the pump and replace the cap.

8.3.8 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the instrument run logbook.

8.3.9 On the AS-90 control window either click on "Run All" to run both standards and samples or "Calibrate" to run only the standards. If you choose only to calibrate at this time, you will need to click on "Reset Sampler" and "Run Samples" when calibration is complete. Analyze standards and samples. Samples that exceed the upper calibration range must be diluted and reanalyzed.

8.3.10 When analysis is complete place both carrier tubing inlet and the reductant tubing inlet with the auto-sampler probe in a beaker of DI water. Allow the water to pump through the system. Continue to flush all the water through until no more bubbles appear in the waste tubing. Turn off the pump.

8.3.11 Turn off the FIMS unit.

8.3.12 No rinse between samples is necessary per manufacturer's instructions.

8.3.13 The following Analytical Sequence should be used.

1. Calibration Blank(S0)
2. Standard #1 (S1)
3. Standard #2 (S2)
4. Standard #3 (S3)
5. Standard #4 (S4)
6. Standard #5 (S5)
7. ICV
8. ICB
9. Method blank
10. LCS
11. Samples (≤6)
12. CCV
13. CCB
14. MS
15. Sample Duplicate
16. Serial dilution
17. Samples (<5)
18. CCV
19. CCB

8.3.14 All information including the analytical sequence is documented in the FIMS 100 Run Logbook, **Attachment 2.**

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of ug/L for aqueous samples, and ug/Kg dry weight for soils/solids. Results are reported to two significant figures.

9.1.1 For aqueous results, report the data generated directly from the instrument with allowance for sample dilution. Upload the data from the FIMS directly into the Omega LIMS system for reporting.

9.1.2 For soil/solid samples, upload the data as for aqueous samples. The LIMS will calculate the final results in soil units, with allowance for dilution, sample weight and percent moisture.

9.2 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)

where: SSR = spiked sample result
SR = sample concentration

SA = spike added

9.3 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10 Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Linear correlation for the standard curve must be **≥0.995**. The Calibration curve must not be forced to go through the origin zero point.

10.3 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method Blanks must not contain Mercury at a concentration ≥ the MRL. If mercury is present in the Method Blank and the lowest sample concentration in the batch exceeds 10 times the blank concentration, no corrective action is needed. Otherwise, corrective action for method blank contamination involves determining the source of the contamination and re-prepping the entire batch.

DoD QSM - Method blank concentrations must be less than 1/2 RL.

10.4 Calibration verification – A second source ICV prepped with the associated samples is analyzed immediately after the curve and must be within 90-110% of its true value. The ICV concentration is at the mid-level of the calibration curve.

10.5 CCVs are analyzed at least every 10 samples with 80-120% recovery requirements.

10.6 An ICB/CCB is run immediately after the ICV/CCV set; the mercury value in the ICB/CCB is not to exceed the MRL.

DoD – Mercury is not to exceed 2 times MDL in the ICB/CCB.

10.7 A matrix spike and a matrix duplicate are prepped and analyzed with every batch not to exceed 20 samples. The RPD for duplicates is 20% and the Aqueous spike recovery control limit is 75-125%. The soil spike recovery control limit is 80-120%.

DoD – Spike recoveries must be within 80-120% for both water and soil

10.8 Laboratory Control Sample (LCS) -An LCS is prepped with a minimum of every 20 samples of the same matrix. Control limits are 80-120% of the true value for mercury. If the LCS is outside the acceptance limits, the corresponding samples are re-prepped and reanalyzed. Corrective action includes re-digestion/reanalysis for all samples and QC in the batch.

10.9 Dilution test (serial dilution) - A dilution test is performed once for each analytical run or if a unique sample matrix is encountered. If the analyte concentration is at least 25 times the MDL, an analysis of a 5 times dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected.

DoD- Serial Dilution is required once per preparation batch.

10.10 Sample concentrations that exceed the highest calibration are diluted and rerun so that their concentration fall within the calibration range.

10.11 The Inorganic Laboratory Supervisor/Manager authorizes any method deviations.

10.12 The intermediate standard is stable for 28 days. All standards made from a primary standard expire on or before the primary standard's expiration date.

10.13 Method detection limits (MDLs) are established once a year. The MDL is obtained by multiplying the standard deviation of seven or eight analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the expected MDL. An MDL verification check is performed immediately following MDL study, and quarterly each year in lieu of the annual MDL study if desired. An MDL verification check sample is spiked at approximately 2 times the current MDL.

10.14 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the maintenance logbook. Trends are to be monitored to show the instrument is within the optimal absorbance range. The normal/optimal absorbance range is from 0.17-0.24.

11. Data Validation and Reporting

11.1 Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the Supervisor/Lab Manager. The Supervisor/Lab Manager reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. After each review, the appropriate section of the Data Review Checklist is checked off, **Attachment 3**.

11.2 Reports are generated by the data reporting group. The data submitted for report preparation is dependent on project requirements.

11.3 Data is always reported to the MRL. If clients require reporting limits different from the MRL/PQL, the data will need to be reported using a special form set through the Omega LIMS system depending on project needs.

11.4 Electronic files (EDD) are generated by the Data Management Department and are stored in the server until downloaded to tapes by the server backup system.

12. Corrective Action Procedures

12.1 Corrective Action to be implemented in the event QC results are outside of the acceptance range are covered in **section 10**.

12.2 Corrective Action reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a corrective action report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. These sheets are stored in the Health & Safety Officer's office. In addition, laboratory personnel

should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric, sulfuric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 The cell-heating compartment maintains a temperature of 100°C throughout the analysis. Care should be taken to avoid burns from the cell-heating compartment.

13.4 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency: Test Methods for Evaluating Solid Waste, Update IIB, SW-846 Method 7470A and Update IV SW-846 Method 7471B.
2. Department of Defense, Quality Manual for Environmental Laboratories. Final Version 3, January 2006.

Attachments:

Attachment 1: Mercury Digestion Logbook
Attachment 2: FIMS Instrument Logbook
Attachment 3: Data Review Checklist Form

Attachment 1
Mercury Digestion Logbook

MITKEM LABORATORIES

Mercury Digestion Logbook

						Reagents Added							
Date	Bottle No.	Sample ID		Client ID	Sample Vol (ml)/ Wt (g)	Conc. H_2SO_4 (ml)	Conc. HNO_3 (ml)	5% $KMnO_4$ (ml)	5% K_2SO_4 (ml)	Aqua-regia (ml)	Final Volume (ml)	Comments	Analyst

Waters
In: _____
Out: _____
Matrix: Aqueous

Soils
In: _____ Out: _____
In: _____ Out: _____
Matrix: Soil/Solid

LCSS ID _____
LCSS volume: _____
Spike ID _____
Spike volume: _____

Temp: _____ °C
H_2SO_4 Lot # _____
HNO_3 Lot # _____
HCl Lot # _____
KMnO4 ID # _____
K2S2O8 ID# _____
Method # _____

RELINQUISHED TO: _____

Reviewed by: _____

Logbook ID: 100.0128 - 10/08

Attachment 2
FIMS Instrument Logbook

MITKEM LABORATORIES: FIMS 100 RUN LOGBOOK

Date: **Analyst:**

Analysis:				Analysis:				Analysis:				Analysis:			
SEQ#	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**	
1				26				51				76			
2				27				52				77			
3				28				53				78			
4				29				54				79			
5				30				55				80			
6				31				56				81			
7				32				57				82			
8				33				58				83			
9				34				59				84			
10				35				60				85			
11				36				61				86			
12				37				62				87			
13				38				63				88			
14				39				64				89			
15				40				65				90			
16				41				66				91			
17				42				67				92			
18				43				68				93			
19				44				69				94			
20				45				70				95			
21				46				71				96			
22				47				72				97			
23				48				73				98			
24				49				74				99			
25				50				75				100			

Calibration/ICV Prep Date:_____ SnCl2 _____ HCl: _____

Reviewed By: _____ NaCl: _____ Hydroxylamine Sulfate: _____

Logbook ID: 100.0164-05/08

Attachment 3
Data Review Checklist Form

Mitkem Laboratories,
A Division of Spectrum Analytical, Inc.
Batch Data Review Checklist - Inorganics

Analysis:_____

Level 1 (Analyst) Review:_____ Level 2 (Technical) Review:_____

Workorder:_____

	Yes	No --List/Explain any Unusual Occurrences or Nonconformances
Calibration Acceptable *ICAL / CCAL*		*List all non-conforming project analytes*
LCS Acceptable		*List all non-conforming project analytes*
Blank Acceptable		*List all non-conforming project analytes*
Spike Acceptable		*Reasonable recovery / Matrix effect / Spike to sample concentration ratio*
Dup Acceptable		*Reasonable precision / Sample non-homogeneity?*
Serial Dilution		*List all non-conforming analytes, Matrix effect?*
Post-Digestion Spike		*Matrix effect?*
Within Holding Time		*List runs/re-runs out of holding time; Explain*
Within Analysis Range		*Dilutions properly noted; Explain any "E" flag analytes or dilutions with no target hit*
Calc/Data Xfer Check		*Including proper significant figures, rounding*
Reasonableness Check:		*Compared to historic or on-going trends, or for this analysis in general?*

Notes:

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C

Rev. 12

	Signature	Date
QA Director:	Sharyn B Lauh	2/3/09
Lab Director:	Kin Chui	2/3/09

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil
by Inductively Coupled Argon Plasma Atomic Emission Spectrometry
using Method SW846 6010C
Rev. 12

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the elements listed in **Attachment 1**. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. **Section 8.1** provides the Method references for sample digestion procedures. See **LIMS Test Information/ Test/Limits** for analytes and their associated MDL/PQLs.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, samples must be digested using appropriate sample preparation methods.

3.2 The method measures element specific emitted light by optical spectrometry. The samples are nebulized and the resulting aerosol is transported to the plasma torch. The metals pass through the hot zone of the plasma, where they take up energy. Subsequently the metals pass through the cold zone (relatively) of the plasma where they give up the excess energy at element specific wavelength. The spectra are dispersed by a grating spectrometer, and the intensity of the emitted light is measured by a solid state photomultiplier. Background correction is required for trace element determination. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the

analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For metals analysis by Method 6010C, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Soil samples are stored at 4°C ± 2°C until analyzed.

4.3 Sample holding time for metals analysis by method 6010C is 180 days from the date of sample collection for both water and soil.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICAP-AES.

5.1 Spectral interferences - Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena. The first of these can be compensated for by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. The second effect may require selection of an alternative wavelength. In addition one could select an alternate wavelength where interference is minimal or absent. The 4300DV and the 3100XL used at Mitkem have many spectral lines from which to choose. The third effect can usually be compensated by a background correction adjacent to the analyte line.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of

operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer, or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.5 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. If interference caused by a particular sample matrix is known, in many cases it can be circumvented. However, when the nature of the sample is unknown, following tests can be used to ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.5.1 Dilution Test -If the analyte concentration is sufficiently high (minimally a factor of 10x above the method detection limit (MDL)), an analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect could be suspected.

5.5.2 Post Digestion Spike Addition - If the matrix spike (pre-digestion) recovery falls outside of the control limits (75% - 125%), matrix interference is suggested. In this case an analyte spike is added to a portion of a prepared sample or its dilution at a level just below the mid-point of the calibration curve. Post digestion spikes should be recovered to within 80% - 120% of the known value. If not, a matrix effect should be suspected.

5.5.3 Comparison with alternative method analysis-when investigating a sample matrix, comparison tests may be performed with other analytical techniques, such as atomic absorption spectrometry, or ICP-mass spectrometry. This should only be done after consultation with the client.

5.5.4 Internal Standard Addition technique can be used (Refer to Section 4.4.2 of Method 6010C.)

6. Equipment and Apparatus

6.1 Inductively coupled argon plasma emission spectrophotometer (ICAP).

The ICAPs used at Mitkem are a Perkin-Elmer Model 4300DV and a 3100XL. The 4300DV is outfitted with an AS-93plus, 157-position autosampler and a high precision, three channel, peristaltic pump. The 3100XL is outfitted with an AS-91, 160-position

autosampler and a high precision, three channel peristaltic pump. Both ICPs have axial viewing capability, as compared to the more traditional radial viewing ICAP. Axial viewing provides greater sensitivity for all elements analyzed. The solid state detector is capable of analyzing at approximately 6000 wavelengths.

The built-in radio-frequency generator is FCC compliant.

The systems are computer controlled through a 32-Bit, Microsoft Windows NT operating system. This system allows for great flexibility in controlling the instrument.

The required high purity argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.1.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. Instrument detection limits, linear dynamic ranges, and interference effects are established for each analyte line used. All measurements must be in the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

6.1.2 Daily emissions of the highest standard for arsenic, copper, lead and selenium are recorded as a means to ensure the plasma is stable before analysis and also to chart potential problems especially with the power tube, sample introduction system, or RF generator. The normal range of the four analytes above are (the unit is intensity generated by instruments):

Optima 2 (3100 XL)	Optima 3 (4300 DV)
As 2,000 - 3,000	As 2,000 - 2,500
Cu 600,000 - 900,000	Cu 400,000 - 600,000
Pb 12,000 - 20,000	Pb 4,000 - 8,000
Se 2,000 - 4,000	Se 1,800 - 2,400

6.1.3 For analysis of normal environmental samples use the following standard operating conditions:

Wattage: 1450 – 1500
Argon flow rate (L/min): 15
Nebulizer flow rate (L/min): 0.55
Sample flow rate (mL/min): 2
Rinse time (sec): 60
Read delay (sec): 60

6.1.4 For regular day-to-day ICAP measurements, the plasma need not be optimized prior to analysis. The plasma needs to be optimized each time a major change occurs in instrument configuration, such as replacement of the torch and/or maintenance, unless response is still within normal ranges. Responses and alignment are routinely checked after torch cleaning or replacement with the analysis of a 1 mg/L Mn solution for axial view calibration and 10 mg/L Mn solution for radial view calibration. "Optimization" is a function of a series of procedures; refer to the Mitkem SOP 100.0006 for additional information on how to perform general maintenance and troubleshooting procedures.

6.1.5 To optimize the plasma, follow the instructions in the Optima Software Guide.

6.2 ICAP Instrument Maintenance:

6.2.1 Preventative Maintenance:

- Peristaltic pump tubing will be replaced every 16 hours of instrument time or sooner if memory effects are manifested.
- The plasma torch is cleaned using concentrated HNO_3 when torch and sample injector buildup is noted.
- The spray chamber and nebulizer are cleaned approximately every month as needed. Replacement is done when needed.
- Air filters are cleaned once every two weeks or as needed upon visual inspection.
- The RF coil, window and cone are cleaned every 2-3 weeks.
- The instrument undergoes extensive maintenance by a manufacturer's service engineer as needed.

6.2.2 Troubleshooting:

6.2.2.1 Sudden drop in CCV concentration occurs in a run:

- Check to see that sample introduction system (tubing) has not become disconnected.
- Check to see if a clog has occurred in the sample introduction system.
- Check CCV Standard to ensure it is not empty.
- Check room temperature to see if a fluctuation has occurred.

- Recalibrate and rerun if necessary.

6.2.2.2 CCVs drift up and down:

- Remake the CCV standard.
- Check room temperature to see if a fluctuation has occurred.
- If problem persists, call Perkin-Elmer. The problem is usually indicative of a power tube failure.

6.2.2.3 Plasma goes out during analysis:

- Sample is not reaching spray chamber (tubing came apart or there is a clog)
- High levels of salt in the sample caused plasma temperature changes.
- The sample injector is coated with high salt material and needs to be changed.
- The power tube is going (after all others fail repeatedly) and Perkin-Elmer must be called for service.

6.3 Glassware:

6.3.1 Class A volumetric flasks: 100 mL and 50 mL.

6.3.2 Class A volumetric pipettes:

6.3.3 10-100µL adjustable Eppendorf.

6.3.4 100-1000 µL adjustable Eppendorf.

6.3.5 5mL adjustable Fisher Scientific.

6.3.6 100µL and 1mL fixed Wheaton.

6.3.7 10 mL Wheaton ICP tubes.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, Accustandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. ***Please note that standards and reagents from other vendors could be***

used as long as the standards are of high purity (>96%0 and traceable to reference materials. All standards are labeled as instructed in Mitkem SOP # 80.0001 Standard Preparation, Equivalency and Traceability.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.

7.2 Nitric acid (conc.), HNO_3, Trace Grade, Fisher Scientific.

7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Mixed calibration standard solutions. Mixed calibration standards, obtained from High Purity Standards, come in three stock solutions. Calibration standards and QC solutions are made up on a daily basis.

7.4.1 CLP CAL 1 consists of two solutions:

- CLP CAL 1B contains silver only.
- CLP CAL 1A contains all other elements other than antimony, arsenic, cadmium, lead, thallium, and selenium.

7.4.2 CLP CAL 2 contains antimony only.

7.4.3 CLP CAL 3 contains arsenic, cadmium, lead, thallium, and selenium.

7.5 High standard (**ICP Standard 1**):

- Pipet 1mL HNO_3 (conc) and 1.5mL HCl (conc) into a 100mL volumetric flask.
- Pipet 1mL each of CLP CAL 1A & 1B stock solution and 0.1mL each of intermediate CLP CAL 2 and 3 into the flask.
- Pipet 0.1mL of Titanium single element standard into the flask.
- Bring to volume with DI water.

7.6 Second (middle) standard (**ICP Standard 2**):

- Pipet 5mL of a 1% HNO_3 and 1.5% HCl acid mixture into a 10 mL ICP tube.
- Add 5mL of the ICP Standard 1.

7.7 Third (low) standard (**ICP standard 3**):

- Pipet 10mL of 1% HNO_3 and 1.5% HCl solution into a 10 mL ICP tube.
- Withdraw 100µL.
- Add 100µL of ICP Standard 1 and mix well.

See **LIMS Test Information/ Test/SPEC** for concentrations of the Initial Calibration Standards.

7.8 **Second source** ICV/CCV standards (CCV100XCONC and Antimony (Sb) Stock solutions) are obtained from Accustandard.

- Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl (conc.) into a 100mL volumetric flask.
- Pipet 1.5mL (for ICV)of CCV100XCONC stock solution into the flask.
- Pipet 0.15mL of Antimony (Sb) Stock solution into the flask.
- Bring to volume with DI water.
- Transfer the solution to a 50mL plastic ICP tube.
- This standard is prepared as needed (in amounts consistent with the ratios above) usually every 1 – 2 days.

See **LIMS Test Information/ Test/SPEC** for analytes, concentrations and recovery limits for the ICV/CCV.

7.9 QC Standards ICSA and ICSB solutions are obtained from High Purity Standards.

ICSAB solution: In addition to the normal element composition of the ICSA solution, sodium, potassium, lead and selenium are added to the ICSAB solution. These stock solutions are also obtained from High Purity Standards.

7.9.1 Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl into a 100mL volumetric flask.

7.9.2 Pipet 1mL of ICSB stock solution (ANALCS-R) into the flask.

7.9.3 Pipet 10mL of ICSA (CLP-INF-1) stock solution into the flask.

7.9.4 Pipet 0.5mL of the sodium/potassium stock solution (INFCS-5) into the flask.

7.9.5 Pipet 1mL of 45mg/L Pb intermediate standard solution into the flask.

7.9.5.1 The intermediate is prepared by adding 4.5mL of Pb at 1000mg/L (#100028-1) up to 100mL with 1%HNO_3 and 1.5% HCL acid solution in a volumetric flask.

7.9.6 Pipet 1mL of 45mg/L Se intermediate solution into the flask.

7.9.6.1 The Selenium intermediate standard is prepared by adding 4.5mL of Se at 1000mg/L (#031) up to 100mL with 1% HNO_3 and 1.5% HCl acid solution in a volumetric flask

7.9.7 Bring to volume with DI water.

7.9.8 Transfer the solution to a 10 mL ICP tube.

See **LIMS Test Information/ Test/SPEC** for concentrations of the ICSA and ICSAB.

7.10 Laboratory control samples (LCS) and Matrix spikes: The LCS standards for soils and waters are obtained from High Purity Standards.

- LCS/Spike standard 1 is prepared by adding 455uL of CLP-CV-1 to the digestion tube.
- LCS/Spike standard 2 and 3 are prepared by adding 45.5uL each of CLP-CV-2 and CLP-CV-3 to the digestion tube.

See **LIMS Test Information/ Test/SPEC** for final concentrations of the LCS/MS.

7.11 Linear Dynamic Range

The stock linear dynamic range (LDR) standards are purchased from Ultra Scientific, Inorganic Ventures and High Purity. The LDR is determined as the highest concentration of standard in which the determined value is within 10% of the true value.

- *LDR Standard I* is prepared by addition of 1 mL of Pb at 1000 mg/L, volumized to 10 ml with 1% HNO3 and 1.5% HCl. Final concentration 100mg/L.
- *LDR Standard II* is prepared by addition of 5 mL of mix QCS-19 and 0.5mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 50 mg/L.

- *LDR Standard III* is prepared by addition of 2.5 mL of mix QCS-19 and 0.25 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 25 mg/L.

- *LDR Standard IV* is prepared by addition of 1.0 mL of mix QCS-19 and 0.1 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 10mg/L.

- *LDR Standard V* is prepared by addition of 0.5 mL of mix QCS-19 and 0.05ml of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 5mg/L.

- *LDR Standard VI* is prepared by addition of 0.5 mL of Al and 0.5ml of Mg at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard VII* is prepared by addition of 3.0 mL of Fe at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 300mg/L.

- *LDR Standard VIII* is prepared by addition of 0.5 mL of Ca at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard IX* is prepared by addition of 0.5 mL of mix INFCS-5 (High Purity) and volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 250mg/L.

8. Procedure

8.1 The methods in SW-846 for sample digestion or preparation are as follows:

8.1.1 Method 3005 , SOP No.100.0003, prepares ground water and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with hydrochloric and nitric acids prior to metal determination.

8.1.2 Method 3010 , SOP No.100.0003, prepares aqueous samples, mobility-procedure extracts and waste samples that contain suspended solids for total metal determination by ICP. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

8.1.3 Method 3050 , SOP No.100.0104, prepares solid waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrochloric acid.

8.2 The wavelengths and background correction locations in the reference method can be substituted if they can provide the needed sensitivity and are corrected for spectral interferences. The analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

For each analyte there are a number of possible wavelengths at which analyses could be made. The wavelengths used were selected based on consultations with the ICP specialists at Perkin-Elmer and our own experience.

8.3 Background correction factors are obtained by aspirating a concentrated solution, usually about 200-500ppm, of the interfering element, and measuring the resulting concentrations at the wavelength used. The background correction factors for that interfering element are obtained by dividing the measured concentrations by the actual concentration. For example: by aspirating a 500ppm solution of aluminum, one measures a cadmium concentration 0f 0.010ppm. The aluminum correction factor for cadmium is (0.010)/500 = 0.000020. This means that if the aluminum concentration in a sample is, say, 120ppm, one must subtract 120*0.000020 = 0.0024ppm from the measured cadmium concentration in that sample.

8.4 Interelement Correction (IEC) factors are established quarterly (or biannually at a minimum), for the major salts: aluminum, calcium, iron, magnesium, and also for chromium, copper, manganese, nickel, thallium, titanium and vanadium. Correction factors should not exceed 20% difference from the previous IEC values.

8.5 Linear Dynamic Range is performed quarterly (or biannually at a minimum). Following instrument calibration, solutions with varying concentrations (**section 7.5.13**) of each analyte (LDR standards) are analyzed. The highest concentration, within ±10 % of the true value, establishes the linear range.

8.6 Allow the plasma to become thermally stable before beginning the analyses. This usually requires at least 30 minutes. If the plasma is (for whatever reason) extinguished and needs to be re-lit, subsequent re-stabilization of the plasma takes only 15 minutes provided the plasma is re-lit immediately after it is extinguished.

8.7 The nebulizer flow rate used is constant for all aqueous sample extracts and needs not be reset prior to analysis.

8.8 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits (IDL). (See **Section 10** for IDL calculation)

8.9 The calibration curve consists of a blank (S0) and at least 3 calibration standards. The low concentration calibration standard concentration is less than or equal to the method reporting limit (MRL). Sample concentrations less than the lowest standard can only be reported after method modifications have taken place, or with estimated results flagging.

The highest concentration must be within the instrument's linear dynamic range. The standards are run in a sequence from high concentration to low concentration.

8.10 The minimum correlation coefficient for the calibration curve is 0.998. If a correlation coefficient less than 0.998 is obtained, the calibration must be repeated.

8.11 A mid-range ICV (Initial Calibration Verification) Standard from an independent source than the calibration standards) precedes the analysis of the samples. Recovery limits for the ICV are ± 10% of the true value. If the ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. An ICV can be reanalyzed only once prior to corrective action.

8.11.1 A low level ICV (not required to be second source) is run to verify the lower level of calibration . Recovery limits for the low level ICV are + 30% of the true value. The low level ICV is the same concentration as the lowest standard in the initial calibration (S3), which is at the reporting limit. If the low level ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. A low level ICV can be reanalyzed only once prior to corrective action.

8.12 A CCV (Continuing Calibration Verification Standard) is analyzed after at least every tenth sample and at the end of the sample run. Recovery limits for the CCV are ±10% and in the case of failure for a particular element, all samples following the last acceptable CCV must be reanalyzed for that element. A CCV can be reanalyzed only once prior to corrective action.

8.13 The ICB is analyzed after the ICV and is of the same source as the calibration blank. The CCB (Continuing Calibration Blank) is analyzed after each CCV. Concentrations of any analytes detected must not exceed the MRL or corrective action, such as a single reanalysis and evaluation, must be taken. If the ICB/CCB still fails, reanalysis of all samples since the last valid ICB/CCB for that element is required.

DoD QSM: The acceptable absolute value of the ICB and CCB must be < 2x MDL.

8.14 The ICS (ICSA and ICSAB) standards must be run at the beginning, every 8 hours and at the end of each analytical run.

8.14.1 The ICSA solution, containing the Al, Ca and Mg at 500mg/L, and Fe at 200mg/L, must be run at all wavelengths used for each analyte reported. The analytical results for those target analytes with MRLs ≤ 10ug/L shall fall within ± 2x MRL of the analyte's true value (the true value shall be zero unless otherwise stated). If the results for these analytes fall outside the ± 2x MRL window, check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. For analytes with a MRL > 10 ug/L, the ICSA results shall fall with ± one MRL of the analytes true value (0).

DoD QSM: the absolute value of all non-spiked analytes < 2x MDL

8.14.2 The ICSAB contains Al, Ca, Mg and Fe at the same concentrations as the ICSA as well as all other analytes of interest. Recovery limits for the ICSAB must be within ± 20% of the true value. If the ICSAB recoveries do not meet acceptance criteria, corrective action must be taken. Check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. If the ICSAB at the end of an analytical sequence fails for a particular element, all samples must be reanalyzed for that element.

8.15 After completion of the initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine. A 60 second rinse (rinse solution: 1% HNO_3 and 1.5% HCl) is conducted between all sample solutions, quality control samples, method blanks, and standards.

Analytical Sequence: The following QC protocol should be employed.

1. Standard S0
2. Standard S1
3. Standard S2
4. Standard S3
5. mid-range ICV(second source)
6. low-level ICV (either source)
7. ICB
8. ICSA
9. ICSAB
10. Sample 1
11. *
12. *
13. *
14. *
15. *
16. *
17. Sample 8
18. CCV
19. CCB
20. Sample 9
21. *
22. *
23. *
24. *
25. Sample 16
26. ICSA
27. ICSAB

28. CCV
29. CCB

Note: The ICSA and ICSAB sample count as analytical samples between CCV/CCBs.

Any deviations must be approved by the Inorganic Laboratory Manager or the Operations Manager before they can be implemented.

All analyses are documented in the Instrument Run Log (**Attachment 3**) .

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Results are reported to two or three significant figures.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the reporting limit (RL) unless specifically requested by the client.

9.3 Soil concentrations are calculated using the equation below:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times Df}{W}$$

Where:
C = Concentration in extract (µg/L)
V = Volume of extract (L, 100mL = 0.1L)
Df = Dilution factor (undiluted =1)
W= Dry weight of sample aliquot extracted (g)

9.4 Recovery Calculations:

The recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)

Where:
SSR = spiked sample result
SR = sample concentration
SA = spike added

9.5 Relative Percent Difference Calculations:

The relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100\%$$

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (replicate)

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method blank concentration must be less than or equal to reporting limits unless the sample concentration is at least 20 times greater than the blank concentration or less than reporting limits. Corrective action for method blank contamination involves determining the source of the contamination and re-prepping the affected samples in the batch. The analyst may rerun the method blank once as the first step of corrective action.

DoD QSM -Method blank concentrations must be less than or equal to one-half the reporting limit. For common contamination analytes, method blank concentration must be less than or equal the reporting limit.

10.3 Calibration verification –

10.3.1 A mid-range ICV analyzed immediately after standards must be within ±10% of the true value. The ICV is an independent source standard purchased from a different vendor than the calibration standards.

10.3.1.1 Method 6010C recommends an additional calibration verification using a low-level ICV with a 70-130% criteria, however only the mid-range ICV is required to be from an independent source. In lieu of a second analysis, the response from the S3 standard can be entered back into the curve for verification.

10.3.2 The CCV is analyzed a minimum of every 10 samples in the analysis and at the end of the analytical sequence. If the closing CCV does not meet the criteria, the CCV and all analyses from the opening CCV must be re-analyzed after the problem has been eliminated.

10.3.3 The ICV and CCV analyses are followed by the ICB and CCB analyses, respectively. The ICV and CCV must pass the ±10% criteria or be re-analyzed.

10.4 Use of all standards made from a primary standard must not exceed the primary standard's expiration date.

10.5 Matrix spike (MS) samples - A matrix spike is processed with each batch of samples. Spike recoveries must be within 75-125% of the expected value. If the native sample results exceed 4x the spike added, no further action is needed. Note in narrative. Unless superceded by project requirements, it is not necessary to spike Na, K, Ca, and Mg for waters; or Na, K, Ca, Mg, Fe, Al and Mn for soil.

DoD QSM: Spike recoveries must be within 80-120%. Precision requirements are ≤ 20 % RPD for both aqueous and soil matrices. There is no corrective action for MS recoveries outside the acceptance range other than data qualification.

10.6 Matrix duplicate (DUP) or matrix spike duplicate (MSD) samples (Both options are allowed in method SW6010)- Duplicates are prepped and analyzed with every batch not to exceed 20 samples. Relative Percent Difference (RPD) is calculated for the results of duplicate samples.

10.6.1 A limit of 20% RPD shall be used for sample values greater than 5x the MRL

10.6.2 A control limit of (±) the MRL level must be used if either the sample or the duplicate value is less than 5x MRL.

10.6.3 If one result is above 5x MRL and one below, use the ± MRL criteria.

10.6.4 If both values are below the MRL, no RPD is calculated.

DoD QSM- DUP precision is evaluated for all analytes. Precision requirements are ≤ 20 % RPD for both water and soil. There is no corrective action for MS/MD precision outside the acceptance range other than data qualification.

10.7 Laboratory Control Sample (LCS) - is prepped with a minimum of every 20 samples of the same matrix.

10.7.1 For an aqueous LCS sample, mixed standards are spiked into a beaker of DI water resulting in concentrations approximately ½ the concentration of the high calibration standard and prepped as an aqueous sample. Recoveries must be within the established control limits. The ID of the aqueous LCS sample is LCSW.

10.7.2 For soils, approximately 1g of Teflon Chips (Chemware Ultra-pure PTFE boiling stones, acid washed) used to simulate solid matrix is spiked with standards at the approximate mid-point of the calibration curve. The LCS is then prepped as a soil. The ID for the solid LCS sample is LCSS.

DoD QSM –The acceptance range for both water and soil is 80-120% except silver which has a 75-120% range for soils. When Mitkem in-house LCS limit fall within DoD limit, in-house limits may be reported. If Mitkem in-house limits are wider than DoD limits, the 80-120 % limits must be used.

Laboratory control sample acceptance limits are based on control charts, which are established at Mitkem. These are referred to as in-house limits. Mitkem may also adopt limits from outside agencies when necessary.

10.8 Post digestion spike (PDS) addition - An analyte spike added to a portion of the sample digestate. A PDS is analyzed with each preparation batch of samples (PDS spiking of the same sample as used for matrix spiking is recommended) and is suggested when a new or unusual matrix is encountered within a batch. The acceptance criteria for PDS is 80-120%. The PDS is spiked at the same concentrations as the matrix spike. The PDS concentration should be >10 times the MRL, but < 100 times. If the PDS is not recovered within the specified limits, a matrix effect is confirmed.

DoD QSM: A PDS is required when a new or unusual matrix is encountered within a batch; or when the serial dilution test fails. In addition, a PDS is recommended for projects where all samples are below 50X MDL. The acceptance window is 75-125%.

Corrective action should be taken if the spike concentration <u>is at least 2x</u> the native sample concentration:

(a) If the MS fails for any analyte but the PDS passes, the sample may be redigested and analyzed, except when low MS recovery can be explained as a historically poor performer (example:Antimony (Sb) for soils) in that matrix. In these instances the analyst may want to discuss the outliers with the project manager or department supervisor. When redigestion is warranted, and the MS fails in the redigestate, a matrix issue is confirmed. If the MS passes for the redigestate, the initial results were due to human error during digestion or spiking.

(b) If both the MS and PDS fail, the laboratory will perform a dilution test, or may make a reasonable effort to address the matrix interference by performing the method of standard additions (MSA) or internal standard addition.

10.9 Dilution test (Serial Dilution) – should be performed with each preparation batch. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the MDL), an analysis of a 5X dilution should agree within ±10% of the original determination. If not,

a chemical or physical interference effect is confirmed. A serial dilution should be run whenever the PDS fails, to confirm matrix effect.

DoD QSM: A Serial Dilution is required once per preparation batch or when an unusual sample matrix is present. If the SD fails, a PDS must be run.

As a general rule, Mitkem performs a PDS and SD with each preparation batch of samples.

10.10 Instrument detection limits (IDLs) are established at the time the instrument is set up and every six months thereafter. Ten solutions of diluted acid (1% HNO3 and 1.5% HCl) are analyzed on three non-consecutive days. The IDL for a particular element is three times the average standard deviation of the measured concentration for that element. An IDL is valid in the condition that IDL on both instruments are less than MDL currently reported.

DoD QSM: IDLs are established at the time the instrument is set up and whenever significant changes are made

10.11 Method detection limits (MDLs) may be established once a year. Mandatory MDL determination has been removed from the SW846 6010C method however until regulatory agencies are in agreement, Mitkem will continue to perform MDL studies. The MDL is obtained by multiplying the standard deviation of seven analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the calculated MDL. An MDL verification check is performed immediately following the MDL study. In lieu of an annual MDL study, the MDL verification can be run quarterly on each instrument. The MDL verification check sample is spiked at approximately 1-4 times the current MDL and prepared as if it were a sample.

10.12 Sample Dilutions: When analytes exceed the established linear dynamic range, the sample must be diluted sufficiently to bring those elements into the linear range. The dilution factor (Df) must be integrated into the final concentration calculation.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory manager reviews 100% of the data prior to report generation. The QA Director or designee randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 Electronic files are validated by the analyst and transferred to the report generation group via the LIMS. Once data is in LIMS it is reviewed and validated again by the supervisor or his/her designee. The data are then locked and forms may be generated.

11.3 Reports are generated by the reporting group in conjunction with the metals lab. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective action to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated through the LIMS in the event of an out-of -control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel on the bookcase between the GC/MS and OPrep labs. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan, and have read the Mitkem Contingency Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's Quality Assurance Plan.

15. References

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U.S. Environmental Protection Agency. I*nductively Coupled Plasma Atomic Emission Spectroscopy* Method 6010C, SW-846 test methods for evaluating solids waste, Revision 3, Final Update IV, February 2007.

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Attachments:

1. **Attachment 1**: Analytes of Interest (Table 1 from SW-846 6010C)
2. **Attachment 2:** DoD QC Requirements, Table B-6,D-18, D-19
3. **Attachment 3**: Standard Preparation Logs
4. **Attachment 4**: Instrument Run Logbook

Attachment 1
Analytes of Interest

TABLE 1

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678 x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227 x2	17
Molybdenum	202.030	5.3
Nickel	231.604 x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980 x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856 x2	1.2

TABLE 1
(continued)

[a] The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity. Other wavelengths may be substituted (e.g., in the case of an interference) if they provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

[b] The estimated instrumental detection limits shown are provided for illustrative purposes only. Each laboratory must determine IDLs and MDLs, as necessary, for their specific application of the method. These IDLs represent radial plasma data and axial plasma IDLs may be lower.

[c] Highly dependent on operating conditions and plasma position.

Attachment 2:
DoD QC Requirements, Table B-6,D-18, D-19

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18).	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA	

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) (ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within ± 10% of expected value GFAA: within ± 20% of expected value CVAA: within ± 20% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch	No analytes detected > ½ RL For common laboratory contaminants, no analytes detected > RL	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test Serial dilution	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	ICP: Perform post-digestion spike (PDS) addition GFAA: Perform recovery test CVAA: Perform matrix spike	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	The spike addition should produce a level between 10 - 100 x MDL.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x MDL	Recovery within 85-115% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard additions (MSA) or internal standard calibration	When matrix interference is suspected	NA	NA	NA	Document use of MSA in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per preparatory batch per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7470 WATER MATRIX[28]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

[28] The as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7471 SOLID MATRIX[29]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

501L

[29] Some as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

Attachment 3:
Standard Preparation Logs

MITKEM LABORATORIES **METALS PRIMARY STD RECEIPT LOGBOOK:INSTRUMENT LAB**

DATE REC	PRIMARY STD ID	VENDOR	CATALOG#	LOT #	ANALYTE(S)	REC BY	DATE OPENED	EXPIR. DATE	MITKEM ID

100.0038-01/08

Page: _____

MITKEM LABORATORIES, A Division of Spectrum Analytical Inc.

METALS INTERMEDIATE STANDARD LOGBOOK

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Intermediate STD ID	Analyst	Exp. Date

Logbook ID: 100.0041 -01/08

Reviewed By: _____

MITKEM LABORATORIES **METALS WORKING STANDARD LOGBOOK**

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Working STD ID	Analyst	Exp. Date

Logbook ID: 100.0036 -12/07

Reviewed By: _____

Attachment 4:
Instrument Run Logbook

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/4300DV**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 100.0129-05/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/3100XL**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 00.0126-04/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

Mitkem Laboratories,
A Division of Spectrum Analytical, Inc
~~MITKEM CORPORATION~~ SBL 9/22/08

STANDARD OPERATING PROCEDURE

For

Toxicity Characteristic Leaching Procedure

By

SW846 EPA Method 1311

SOP No. 110.025 Rev. 4

	Signature	Date
QA Director:	Karen Gantt	7/13/04
Lab Director:	Edward Jank	7/15/04

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

For

Toxicity Characteristic Leaching Procedure

By

SW846 1311

Rev. 5

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the procedures for the assessment of toxicity characteristics of waste leachate. The Toxicity Characteristic Leaching Procedure (TCLP) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multi-phase wastes. Mitkem routinely performs TCLP on solid samples as well as liquid wastes.

If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the Zero Head Space (ZHE) is not necessary However, the extract from the bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing their work tasks following the procedures outlined in this SOP. Supervisors and managers are responsible for reviewing and approving new and revised SOPs. They are responsible for ensuring that SOPs are accurate, up to date, and are being implemented appropriately.

3. Summary of Procedure

3.1 For liquid wastes (wastes containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8μm glass fiber filter, is defined as the TCLP extract.

3.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. **Note:** Mitkem laboratory does not possess the proper grinding or cutting equipment for surface area or particle size reduction protocols to be followed as stated in EPA SW-846 Method 1311. Most samples encountered by the laboratory are soil samples not requiring particle size reduction. Samples requiring surface area or particle size reduction will be subcontracted to a certified laboratory able to adhere to the Method 1311 specified protocols regarding surface area and/or particle size reduction.

3.3 The solid is extracted with an amount of extraction fluid equal to 20 times the weight of the solid. The extraction fluid employed is a function of the alkalinity of the solid. Following extraction, the liquid extract is separated from the solid by filtration through a 0.6 to 0.8μm glass fiber filter. A special extractor vessel (ZHE) is used when testing for volatile analytes, **figure 2**.

3.4 If compatible (multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected in amber glass jars with Teflon lining.

4.2 Samples are refrigerated at 4°C until time of extraction.

4.3 Holding times for TCLP extraction:

4.3.1 Volatiles:

4.3.1.1 14 days from the date of collection for TCLP extraction.
4.3.1.2 14 days for analysis from the date of TCLP extraction.

4.3.2 Semivolatiles:

4.3.2.1 14 days from the date of collection for TCLP extraction.
4.3.2.2 7 days from the date of TCLP extraction to extract extraction.
4.3.2.3 40 days from the date of extraction to analysis.

4.3.3 Metals:

4.3.3.1 180 days from the date of collection for TCLP extraction.
4.3.3.2 180 days from the date of TCLP extraction to sample analysis.

4.3.4 Mercury:

4.3.4.1 28 days from the date of collection for TCLP extraction.
4.3.4.2 28 days from the date of TCLP extraction to sample analysis.

4.4 Preservatives shall not be added to samples before extraction.

4.5 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

4.6 When the waste is evaluated for volatile analytes, care should be taken to minimize the loss of volatile compounds. Samples must be collected and stored in a manner intended to prevent loss of volatile compounds. Samples should be opened only immediately prior to extraction.

4.7 TCLP extracts should be analyzed as soon as possible following extraction.

5. Interferences and Potential Problems

Potential interferences that may be encountered during analysis are discussed in the individual analytical SOPs (i.e. SOP 90.0012: Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW-846 Method 8260).

6. Equipment and Apparatus

Instrumentation used in this leaching procedure include:

6.1 Tumbler capable of rotating the extraction vessel at 30 ± 2 rpm in an end-over-end fashion, **figure 1**.

6.2 Zero-Headspace Extraction Vessel (ZHE), Millipore, **figure 2**.

6.3 Orbital shaker, VWR Scientific.

6.4 High-density polyethylene (HDPE) bottles. **Please note**; the use of HDPE for the organic extracts is a modification from the method due to the type of agitation apparatus used and breakage of glass containers. The laboratory has documentation showing no cross-contamination of target analytes, blank contamination, or co-elution contamination for blank and Laboratory Control Standard (LCS) documentation.

6.5 pH meter, capable of measuring pH to ±0.05 units.

6.6 Top loading balance, capable of measuring to ±0.1g.

6.7 Gas tight syringe, Hamilton.

6.8 Pressure filter, Millipore.

6.9 Volumetric flask – 1L.

6.10 Graduated cylinder – 10mL, 25mL, 250mL.

6.11 1-L beaker.

6.12 125mL plastic beaker, Nalgene.

6.13 Disposable pipet.

6.14 pH strips, Whatman.

6.15 Borosilicate microfiber filters.

- 90mm, 0.7µm, Advantec MFS, Inc.
- 47mm, 0.7µm, Advantec MFS, Inc.
- 142mm, 0.7µm, Environmental Express.

7. Reagents

The following reagents and gas are used extensively in the lab:

7.1 pH buffers – 4.00, 7.00, 10.00, Fisher.

7.2 DI water.

7.3 Nitrogen gas.

7.4 1N NaOH, Fisher, ACS reagent grade:
40g of NaOH diluted to 1L with DI water.

7.5 Glacial acetic acid, Fisher, ACS trace metals grade.

7.6 Nitric Acid (HNO_3), Fisher, ACS trace metals grade.

7.7 1N HCl, Fisher, ACS trace metals grade:
Dilute 83.3mL to 1L with DI water.

7.8 TCLP Extraction Fluid #1:
Dilute 22.8mL of glacial acetic acid and 257.2mL of 1N NaOH to 4L with DI water. If done correctly, the pH of the fluid will be 4.93 ± 0.05. If pH is not 4.93 ± 0.05, use either 1N NaOH or the glacial acetic acid to bring the pH within that range. The pH of the extraction fluid is documented in the Volatile and/or Non-volatile TCLP/SPLP Extraction Logbooks, **figures 3,4.**

7.9 TCLP Extraction Fluid #2:
Dilute 22.8mL of glacial acetic acid to 4L with DI water. If done correctly, the pH of the fluid will be 2.88 ± 0.05. If not, adjust pH by adding 1N NaOH or glacial acetic acid to bring the pH within the range. The pH of the extraction fluid is documented in the Non-volatile TCLP/SPLP Extraction Logbook.

8. Procedure

8.1 Preliminary Evaluations:

Perform preliminary TCLP evaluations on a minimum 100gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; and (3) determination of which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste.

8.1.1 Preliminary determination of percent solids: percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below. The percent solids information is documented in the Omega LIMS system when required.

8.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) filtration or was previously

determined to be treated as a solid/soil by the project manager and client, proceed to **section 8.1.3**.

8.1.1.2 If the sample is liquid or multiphase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration procedure that is outlined in **sections 8.1.1.3** through **8.1.1.9**.

8.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

8.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

8.1.1.5 Weigh out a sub-sample of the waste (100 gram minimum) and record the weight.

8.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

8.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm to room temperature in the device before filtering. **Note:** if waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in **section 8.1.1.5** to determine the weight of the waste sample that will be filtered. Gradually apply vacuum or gentle pressure of 1 – 10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration. **Note:** instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. **Note:** some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in **section 8.1.1.7**, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

8.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see **section 8.1.1.3**) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in **section 8.1.1.5** or **8.1.1.7**.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (section 8.1.1.9)}}{\text{Total weight of waste (section 8.1.1.5 or 8.1.1.7)}} \times 100$$

8.1.2 If the percent solids determined in **section 8.1.1.9** is equal to or greater than 0.5%, then proceed either to **section 8.1.3** to determine the appropriate extraction fluid or to **section 8.1.2.1** if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids is less than 0.5%, then proceed to **section 8.2.12** if the non-volatile TCLP is to be performed and to **section 8.3** with a fresh portion of the waste if the volatile TCLP is to be performed.

8.1.2.1 Remove the solid phase and filter from the filtration apparatus.

8.1.2.2 Dry the filter and solid phase at 100 ± 20°C until two successive weighing yield the same value within ± 1%. **Note:** caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood.

8.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. Of dry waste + filter) – tared wt. of filter}}{\text{Initial wt. Of waste (section 8.1.1.5 or 8.1.1.7)}} \times 100$$

8.1.2.4 If the percent dry solids is less than 0.5%, then proceed to **section 8.2.9** if the non-volatile TCLP is to be performed, and to **section 8.3** if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the non-volatile TCLP is to be performed, return to the beginning of this section (**8.1**) with a fresh portion of waste, and determine the appropriate extraction fluid (**section 8.1.3**). If only the volatile TCLP is to be performed, see the note in **section 8.1.4**.

8.1.3 Determination of the appropriate extraction fluid; if the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for non-volatile constituents (**section 8.2**), determine the appropriate fluid (**section 7.8** or **7.9**) for the non-volatile extraction as follows:

note: TCLP extraction for volatile constituents uses **only** Extraction fluid #1 (**section 7.8**). Therefore, if TCLP extraction for non-volatiles is not required, proceed to **section 8.3**.

8.1.3.1 Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500ml beaker or Erlenmeyer flask.

8.1.3.2 Add 96.5mL of reagent water to the beaker, cover with a watch glass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is ≤ 5.0, use extraction fluid #1. Proceed to **section 8.2**

8.1.3.3 If the pH is greater >5.0, add 3.5mL 1N HCL, stir vigorously, then check the pH. If the pH is ≤ 5.0, use extraction fluid #1 and proceed to **section 8.2**. If the pH is > 5.0, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.

8.1.3.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to **section 8.2**.

8.1.4 If the aliquot of the waste used for the preliminary evaluation (**sections 8.1.1 – 8.1.4**) was determined to be 100% solid at **section 8.1.1.1**, then it can be used for the **section 8.2** extraction (assuming at least 100 grams remain), and the **section 8.3** extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in **section 8.1.1.7**, then another aliquot shall be used for the volatile extraction procedure in **section 8.3**. The aliquot of the waste subjected to the procedure in **section 8.1.1.7** might be appropriate for use for **section 8.2** extraction if an adequate amount of solid (as determined by **section 8.1.1.9**) was obtained.

The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to **section 8.2.10** of the non-volatile TCLP extraction.

8.2 Non-volatile extraction:

Place 1L high-density polyethylene bottle onto balance and tare. Weigh out a minimum of 100g of sample (solid and liquid phases). In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether Inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

8.2.1 If the waste will obviously yield no liquid when subjected to filtration, weigh out a sub-sample of the waste (100gram minimum) and proceed to **section 8.2.10**. This is the majority of Mitkem's TCLP work.

8.2.2 If the sample is liquid or multiphase, liquid/solid separation is required. This involves filtration and is outlined in **sections 8.2.3** through **8.2.8**.

8.2.3 Pre-weigh the container that will receive the filtrate.

8.2.4 Assemble the filter holder and filter. Place the filter on the support screen and secure. Acid-wash the filter if evaluating the mobility of metals. **Note:** Acid washed filters may be used for all non-volatile extractions even when metals are not of concern.

8.2.5 Weigh out a sub-sample of the waste (100gram minimum) and record the weight. If the sample contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids, use the percent solids information to determine the optimum sample size (100gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

8.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be

decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

8.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, allow the sample to warm to room temperature in the device before filtering. **Note:** if waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in **section 8.2.5** to determine the weight of the waste sample that will be filtered. Gradually apply vacuum or gentle pressure of 10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration. **Note:** instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see **section 8.2.12**) or stored at 4°C until time of analysis. **Note:** some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

8.2.9 If the waste contains <0.5% dry solids, proceed to **section 8.2.12** or **8.2.13**. If the waste contains >0.5% dry solids, proceed to **section 8.2.10**. Quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to **section 8.2.10**.

8.2.10 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{\text{20 X percent solids (section 8.1.1) X weight of waste filtered (section 8.2.5 or 8.2.8)}}{100}$$

Samples considered 100% solid use 100±0.1g of sample and 2000 mL of extraction fluid. Slowly add this amount of appropriate extraction fluid (see **section 8.1.4**) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in the rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of the room in which extraction takes place) shall be maintained at 23 ± 2°C during the extraction period. The rotary device rpm must be checked and the rpms documented in the TCLP Tumbler RPM Logbook.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

8.2.11 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtration through a new glass fiber filter, as outlined in **section 8.2.7**. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed if evaluating the mobility of metals.

8.2.12 Prepare the TCLP extract as follows:

8.2.12.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from **section 8.2.11** is defined as the TCLP extract. Proceed to **section 8.2.13**.

8.2.12.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from **section 8.2.11** with the initial liquid phase of the waste obtained in **section 8.2.7**. This combined liquid is defined as the TCLP extract. Proceed to **section 8.2.13**.

8.2.12.3 If the initial liquid phase of the waste, as obtained from **section 8.2.7**, is not or may not be compatible with the filtered liquid resulting from **section 8.2.11**, do not combine these liquids. Analyze these liquids, collectively defined as

the TCLP extract, and combine the results mathematically, as described in **section 8.2.13**.

8.2.13 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

8.2.14 Compare the analyte concentration in the TCLP extract with the levels identified in the appropriate regulations.

8.3 Volatile extraction:

Use the ZHE device to obtain a TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility on non-volatile analytes. The ZHE device has approximately a 500mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (as defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extract fluid equal to 20 times the weight of the solid phase. Charge the ZHE

with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of sample is not permitted. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any Manipulation of these materials should be done when cold (4^0C) to minimize loss of volatiles.

8.3.1 Pre-weigh VOA vials and set aside. Use a gas tight syringe to express the liquid from the ZHE device.

8.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from **section 8.3**, **section 8.1.1** and/or **8.1.2**). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

8.3.3 If the waste is 100% solid, weigh out 20.0 grams of the waste, record the sample weight and all relevant information in the Volatile TCLP/SPLP Extraction Logbook, and proceed to **section 8.3.11**.

8.3.4 If the waste contains <0.5% dry solids (**section 8.1.2**), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >0.5% dry solids (**sections 8.1.1** and/or **8.1.2**), use the percent solids information obtained in **section 8.1.1** to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

8.3.4.1 For wastes containing <5% solids (see **section 8.1.1**), weigh out a 500 gram sub-sample of waste and record the weight.

8.3.4.2 For wastes containing ≥ 5% solids (see **section 8.1.1**), determine the amount of waste to charge into the ZHE as follows:

% Solids	Weight of waste to charge ZHE*
81 – 99	20.0
61 – 80	25.0
41 – 60	33.3
31 – 40	50.0
5 – 20	100.0

*Mitkem can not use greater than 25.0g sample size due to ZHE internal diameter.

8.3.5 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

8.3.6 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate. **Note:** If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight determined in **Section 8.3.4** to determine the weight of the waste sample that will be filtered. Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

8.3.9 Attach the gas tight syringe to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 10- psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in the 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi , if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container. Transfer the filtrate from the gas tight syringe into the VOA vial. **NOTE:** Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

> **Note:** Some wastes, such as oily wastes and some paint wastes, will obviously contain same material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see **section 8.1.2**), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to **section 8.3.13**. The liquid phase may now be either analyzed immediately or stored at 4°C under minimal headspace conditions until time of analysis.

8.3.11 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (see **section 7.1.4 of SW-846 Method 1311**).

8.3.11.1 Quickly transfer the soil sample to it's corresponding ZHE. Add 400ml of TCLP fluid#1 to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fittings with the square plastic wrench provided. Connect a tank of Nitrogen to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) closed, increase the pressure at the regulator to approx. 20psi. Open the relief valve and begin applying gentle pressure of 10-20psi (or more if necessary) to pressurize the headspace within the ZHE.

8.3.11.2 Place the ZHE device in an exhaust hood and slowly open the liquid inlet/outlet valve to bleed out any headspace created in the ZHE with the addition of the soil sample and extraction fluid. This bleeding should be done slowly and stopped at the ZHE when no more air is observed to be leaking out of the liquid inlet/outlet valve (a small amount of TCLP fluid will also leak out during bleeding). Close the liquid inlet/outlet valve and pressurize the ZHE to about 20-25psi. Check all ZHE fittings to ensure that they are closed tightly. Observe the pressure gauge on the bottom of the ZHE to check for leaks (if the ZHE is not maintaining 20-25psi, check it for leaks as specified in section 4.2.1 of SW-846 Method 1311). If a leak is found, the device should be opened, the sample and fluid disposed of, and the ZHE cleaned. Check the rubber "O" rings in the top and bottom flanges for cracks or misalignment and replace if necessary.

8.3.11.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 +/- 2 °C during agitation.

8.3.11.4 Following the 18 +/- 2 hour agitation period, remove the ZHE's from the rotary agitator. Check the pressure behind the ZHE

piston by quickly opening and closing the gas inlet/outlet valve and and noting the escape of gas.If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in section 4.2.1 of SW-846 Method 1311, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases.

Note: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

8.3.11.5 Connect a tank of Nitrogen to the gas inlet/outlet valve (bottom flange) of the ZHE. At the tank regulator, increase the pressure to about 40psi and open the relief valve. The pressure gauge on the ZHE should read about 40psi also. Attach a 60ml plastic syringe to the liquid inlet/outlet valve (top flange) of the ZHE. Slowly open the liquid inlet/outlet valve and collect the sample fluid in the syringe. When approximately ¾ of the syringe has been filled, close the liquid inlet/outlet valve on the ZHE. Transfer the sample to a clean 40ml VOA vial with no preservative. Repeat this process until two 40ml vials have been completely filled with sample leaving no headspace in the vials. (If an MS/MSD is required for a particular sample six 40ml vials should be collected). Label each vial with it's corresponding Mitkem Lab ID, the TCLP test code, and sample number (1of 2, 2of 2). Store in a VOA refrigerator at 4^0 C until time of analysis.

8.3.11.6 After all blanks and samples have been collected, open ZHE's and collect the remaining TCLP fluid. **DO NOT** pour the remaining fluid down the drain, it's still considered hazardous waste and should be treated as such. Collect all the remaining TCLP fluid and dispose of properly in the Labs liquid waste container. Likewise with the remaining soil and used filter paper. These should also be collected and disposed in the Labs solid waste containers. Completely disassemble and thoroughly clean each ZHE and set aside to air dry.

8.3.12 If the original waste contained no initial liquid phase, the filtered liquid material obtained from **section 8.3.11** is defined as the TCLP extract. If the Waste contained an initial liquid phase, the filtered liquid material obtained from **section 8.3.11** and the initial liquid phase (**section 8.3.9**) are collectively defined as the TCLP extract.

8.3.13 Analyze the TCLP extract according to the appropriate analytical methods. The analytical preparation information is documented in the individual analytical methods SOPs. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

8.3.14 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to **section 10.0** for quality requirements.

9. Data Reduction and Calculations

See Individual Methods.

10. Quality Assurance/Quality Control

10.1 A blank (using the same extraction fluid as used for the samples) must be extracted once for the following, whichever is more frequent:

- Each batch of samples, not more than 20 or
- Whenever samples are extracted.

10.2 A Laboratory Control Standard (LCS) shall be analyzed for each analytical batch.

10.3 A matrix spike shall be performed for each waste type (wastewater treatment sludge, contaminated soil, etc) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, the method quality control criteria will be followed for each analytical method.

10.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Spikes are not added prior to TCLP extraction of the sample.

10.5 The matrix spike concentration should be equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of the TCLP extract as that which was analyzed for the un-spiked sample.

10.6 Matrix spike recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{100\,(X_S - X_U)}{K}$$

Where;

X_S = measured value for the spiked sample.
X_U = measured value for the un-spiked sample.
K = known value of the spike in the sample.

10.7 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the appropriate regulatory level, and (2) the concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

10.8 The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

10.9 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding DI water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

10.10 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standards as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

10.11 Alternately, subtract the instrument signal or external-calibration-derived concentration of the unknown (un-spiked) sample from the instrument signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for the unknowns using the internal calibration curve as if it were an external calibration curve.

11. Data Validation and Reporting

The laboratory supervisors will review and initial all preparation logbook data. This data consist of, but is not limited to, the TCLP Fluids preparation and volumes used, weights of samples, time of extraction, pH documentation, sample and extract handling procedures.

12. Corrective Action Procedures

The corrective action guidelines for the specific quality control criteria specified in the analytical methods must be followed. All out-of-control situations must immediately be reported to the department supervisor or the QA Director. Out-of-control situations must be recorded in the Corrective Action Logbook located in the OMEGA LIMS System, and documented the situation returned to control as described in the Corrective Action Procedures SOP No. **80.0007**.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. MSDS sheets were kept in safety officer's recover. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

US Environmental Protection Agency; "Test Methods for Evaluating Solid Wastes", SW846 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992.

Attachments:

1. **Figure 1**: Rotating Extraction Vessel.
2. **Figure 2**: Zero-Headspace Extraction Vessel.
3. **Figure 3**: Volatile TCLP/SPLP Extraction Logbook
4. **Figure 4**: Non-volatile TCLP/SPLP Extraction Logbook

Figure 1

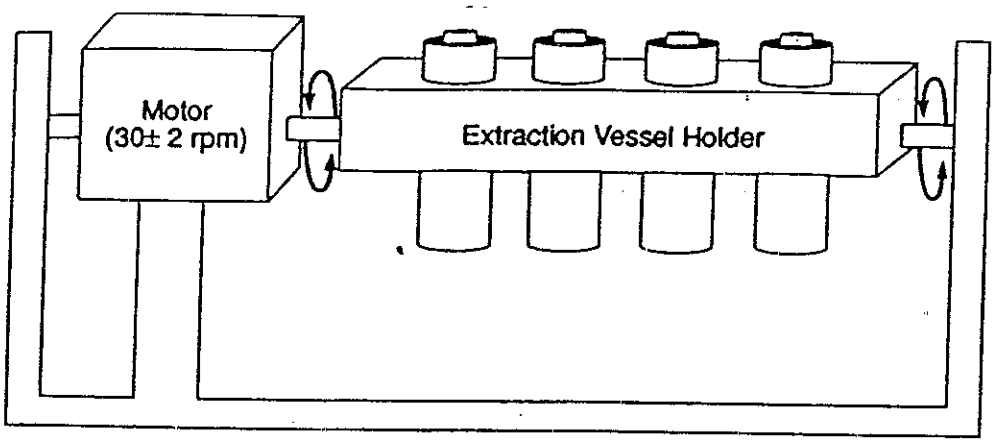

Figure 1. Rotary Agitation Apparatus

Figure 2

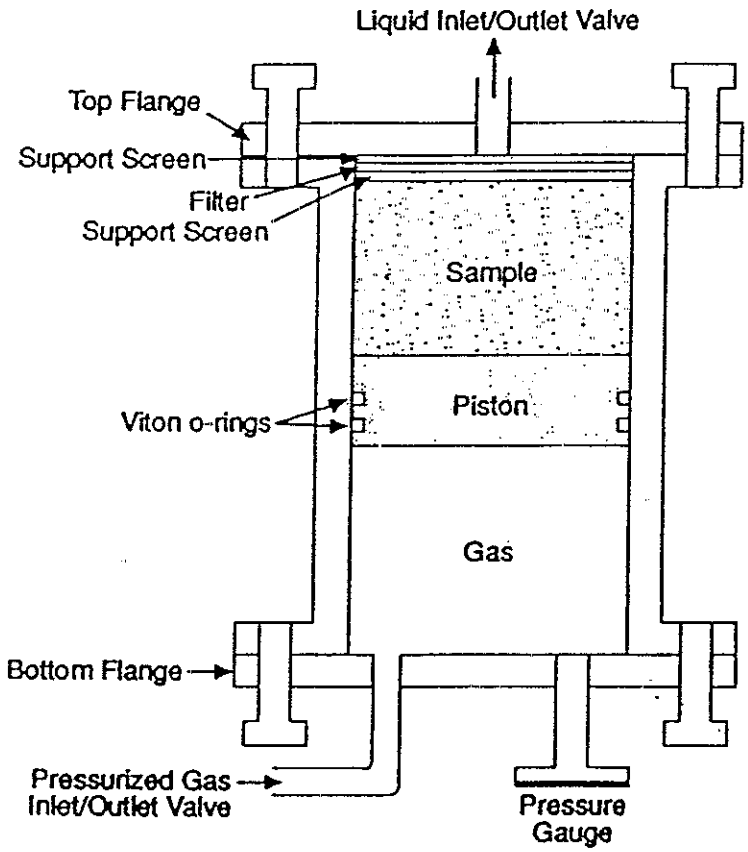

Figure 2. Zero-Headspace Extractor (ZHE)

Mitkem Laboratories

Volatile TCLP/SPLP Extraction Logbook

Extr. Start Date/Time	Lab Sample ID		ZHE #	Extr. Fluid # TCLP/SPLP	Sample Weight	Fluid Volume	Fluid pH	Extr. End Date/Time	FLUID ID# / Comments	Analyst

TCLP Fluid #1 Criteria pH 4.93 +/- 0.05
SPLP Fluid #3 No Criteria

Logbook ID: 90.0075-09/08

Reviewed by _____

FIGURE 3

SOP No. 110 0025 Rev. 5
Date Initiated: 7/22/97
Date Revised: 07/13/04
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MITKEM LABORATORIES

Non-Volatile TCLP/SPLP Extraction Logbook

Start Time/Date	Sample ID		Initial pH	pH After 3.5mL 1N HCl	Ext. Fluid #/ ID	Sample Weight (g)	Fluid Volume (mL)	End Time/Date	Final pH	Metal pH Adj.	Comments	Analyst

SOP No 110.0025 Rev. 5
Date Initiated 7/22/97
Date Revised: 7/13/04
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FIGURE 4

NAOH Lot #_____
HCL Lot #_____
Acetic Acid Lot #_____

TCLP	Fluid #1	Criteria	pH 4.93 +/- 0.05	
	Fluid #2	Criteria	pH 2.88 +/- 0.05	

SPLP	Fluid #1	Criteria	pH 4.20 +/- 0.05
	Fluid #2	Criteria	pH 5.00 +/- 0.05
	Fluid #3	No Criteria	

Logbook ID 110.0171-03/08

Reviewed By: _____

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

for

Handling Evidentiary Materials

SOP No. 110.0026
Rev. 6

	Signature	Date
QA Officer:	Sharon Lauk	2/23/06
Lab Director:	William D.	2/23/06

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

for

Handling Evidentiary Materials

SOP 110.0026
Rev. 6

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the procedures for handling all evidentiary materials associated with a sample delivery group, including air bills, SMO or client traffic report and chain-of-custody forms.

2. Personnel Qualifications and Responsibilities

All employees handling client information should have read and understood this SOP. All information in this SOP must be followed.

3. Summary of Procedure

All paperwork pertaining to the sample delivery group(s) of a case or project must be kept confidential.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Not applicable.

7. Reagents

Not applicable.

8. Procedure

All documents received with a sample delivery group (SDG) and/or generated in the course of the analyses of these samples must be kept confidential. Standard Business Confidentiality practices shall apply.

7.1 Documents specifically marked "Confidential" which may accompany an SDG, must be treated separately from other case-related documents.

7.2 Procedures for handling documents marked "Confidential"

7.2.1 Contact Sample Management Office (SMO) for EPA cases or the client to assure that receipt of these documents is correct and necessary for analysis of the samples. If not required for analysis, return the documents following SMO or client instructions.

7.2.2 If the documents are necessary for sample analysis, place them in a secure file separate from the regular files and under control of the Document Control Officer (DCO) or his/her designee. The DCO will keep this file of confidential documents secure at all times and only allow authorized personnel access on an as needed basis.

7.2.3 Receipt of confidential documentation, its use, duplication and ultimate disposition must be documented in a Confidential Document Log. Duplication of confidential documents must be kept to a minimum and done with concurrence of the Project Officer or his/her designee.

7.2.4 The DCO shall remove and retain for one year the cover sheet of any confidential material disposed of and note the disposition in the Confidential Document Log. Disposal of confidential documents should only be done at the direction of the EPA or client Project Manager and/or the EPA Contracting Officer.

8.4 Test results or data transmitted electronically or by electromagnetic means will be sent in such a way as to preserve client confidentiality. Confidentiality statements are included on Fax forms and e-mail transmissions. Confirmation of client address and/or individual personnel is required prior to transmission.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Not applicable.

11. Data Validation and Reporting

Not applicable.

12. Corrective Action Procedures

Not applicable.

13. Health and Safety

Not applicable.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Not applicable.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

For

Sample Data Control for Inorganic CLP (ILM/ISM)

SOP No. 110.0034
Rev 1

	Signature	Date
QA Director:		3/13/09
Lab Director:		3/16/09

This is a controlled document and may not be copied or written in. Additional copies may be obtained from the QA Department.

MITKEM LABORATORIES,

A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Data Control for Inorganic CLP (ILM/ISM)
Rev 1

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the data management procedures used in support of report generation for CLP samples using the protocols described in ILM04.1, ILM05.4 and ISM01.1.

2. Personnel Qualifications and Responsibilities

Personnel responsible for inorganic data management must be familiar with the protocols outlined in this SOP and are required to follow these procedures.

3. Summary of Procedure

CLP forms 1 – 14 are generated from raw instrument data using the protocols outlined in the CLP Statement of Work (SOWs ILM04.1, ILM05.4 and ISM01.1) and **section 8** of this SOP, in preparation for use in the final data package.

4. Sample Preservation, Containers, Handling, and Storage

NA

5. Interferences and Potential Problems

NA

6. Equipment and Apparatus

6.1 Intel Based Workstation, operating in a Microsoft Windows 05.

6.2 Omega LIMS ILM CLP module (includes ILM, ISM, SW-846 and MCAWW report options).

7. Reagents

NA

8. Procedure

8.1 ThermoFisher PlasmaLab software is used for data acquisition of ICP-MS analyses. Perkin-Elmer WinLab Software is used for data acquisition of all ICP-AES analyses. Once a run has been completed, the data file associated with the run is assigned a filename that describes the instrument, date of run, and the daily run sequence (e.g. A091402 denotes the second data file generated by the Optima 3100 XL (ICP 2) on 9/14). This (.csv) file is loaded onto the file server Bernoulli. Path = \\ Bernoulli \\inorganic\CLPfiles\.

8.2 Subsequent file translation and data processing is done using the Omega ILM Module, Mitkem owns a site license. Currently a copy of the software is kept on three workstations. The workstations are connected to the file server via the Local Area Network (LAN). The servers are located in a secure area accessible by key code entry only to authorized personnel. Details about data storage and backup can be found in Mitkem SOP# 110.0029, Electronic Data Management.

8.3 Once all inorganic data for a Sample Delivery Group (SDG) are generated, a folder containing instrument printouts of all raw data associated with the SDG are delivered to a data report analyst for processing. ICP, Mercury, and Cyanide runs are captured and saved for direct upload by the report generation software.

8.4 The first step in generating a final report begins with the uploading of all runs into Omega LIMS. Before doing this the runs need to be uploaded into Omega Upload Tool so that they can be edited for any problems associated with the run, such as typographical errors, or the "z-ing" out of unwanted data points.

8.5 Enter the runs into Omega LIMS. This is done by opening the Omega LIMS program. At the main menu screen click on the DATA ENTRY button. this will bring up a new screen called Analytical Runs. In this screen enter in new runs, and update or revise any previous analytical runs.

8.6 To enter analytical runs press the Add key in the top tool bar so that there will be a blank screen. Enter the Instrument ID, Run Start Date, Analyst, and any comments. In the comments space enter any information that is important to the run and who uploaded it.

8.7 Once this information is entered press the Data Import button to bring the runs into LIMS. Once the run is uploaded it can be further edited for items such as ID's, turning off analysts that are not reported with the SDG, etc. Then the sequence can be calculated to get the final results.

8.8 The next step is to have the runs that were uploaded into Omega LIMS QA validated by an analyst who has QA authority. ***All runs that are entered into Omega LIMS have to be QA validated before entering them into <u>any</u> CLP module.***

8.9 After the runs have been QA'd by a senior analyst they can be uploaded into the ILM CLP module.

8.10 Open the module and enter an SDG number. Press return after the software has completed some querying.

8.11 Select the protocol that will be used <u>from the protocol drop down list</u>. If the protocol is SW6010<u> or 6020</u> select SW846. If the protocol is <u>CLP</u>, select ILM04.1, ILM05.<u>4 or ISM01.1 as appropriate</u>. <u>If the protocol is E200.7 or E200.8, select MCAWW. This same logic should be applied to the cyanide and mercury fractions as well.</u>

8.12 The next step for ILM04.1 SDGs is to select the IDL set that will be used. To do this left click in the blank IDL Key and a dropdown list will appear. Select the instruments that were used. Once the instruments for the IDLs have been selected, edit the IDLs to the analytes that are associated with the SDG that is being generated.

8.13 Click the "Get LIMS Data" button. This will do many calculations and put all the data on the correct CLP forms.

8.14 Now go to the "Review Data " tab and click on "Update Forms" This will update all the forms that were generated. In this screen the runs can be edited further by deleting the samples that are not needed in the SDG that is being worked on. The analytes that are not needed for the QC samples can be turned off. After all the runs have been edited click the "Update Forms" button again. This icon should be clicked after edits to the run have been made.

8.15 Now go to the tab "Report Options". This lists all the CLP forms that are generated through the module. Click on the icon "Edit Cover Letter" this will update the SDG cover page. Go through all of the forms to make sure that they look right and all the concentrations and percent recoveries appear to meet criteria. To view the forms click in the box next to the form that you want to view then press the "Preview Report". Form 11 ICP Interelement Correction should not be opened with the "Preview Report" icon. The reason for not opening this form is because it will not display properly so just go to "Generate Report" to print it out. After reviewing all the forms go back to the "SETUP: IDL and ICF" tab to import the IEC's. Click on the "Import from Excel" icon this will bring you to a directory where you can navigate to where IEC files are stored. The file is called "zzzzz6010_ICF". Next open the IEC table by clicking on "View Export" button and edit the list by choosing the instruments that are being used. The IEC's can also be uploaded after choosing the IDL's.

8.16 All ILM/<u>ISM</u> SDG's Form 1's need to have the color, clarity, and comments added to then you click on the button "Forms 1 Color, Clarity and Comments".

8.17 Now the report can be generated. Click on the button "Select All". This checks off all the forms that will be printed. If you do not need a form just click off the checkmark then press the "Generate Report" button. This will print out the entire SDG.

8.18 Initial Report Assembly:

Once all data forms have been generated, the entire data package is assembled for final review and contains all the required supporting forms including: all raw instrument data runs, copies of each preparatory log sheets, percent moisture log sheet (if required), a current revision of the laboratory work order that documents the EPA sample names, analyses required and other pertinent SDG related information, appropriate separator pages, and the "CLP/CLP-Like delivery checklist for inorganic analyses"(**Figure 1**). All deliverables shall be presented to the EPA as described in **Exhibit B, Section 2** of the ISM01.1 SOW.

8.19 Final Data Report Review:

Upon receipt of the data package and other supporting documents required in the complete SDG file (CSF), a designated data analyst reviews the final report. This review ensures that all required QA/QC operations as specified in **Exhibit D** of the ISM01.1 SOW have been followed. Errors in the format or content of the final data report are noted and the report is returned to the analyst who originally processed the data for correction.

8.20 Package Assembly:

When the Review/Corrections to the data report have been completed, and approved, the package with all the supporting documentation is delivered to Data Reporting for assembly into the final CSF with the reports (if required) from the organic laboratories. ISM reports also require the DC-2 forms (**Figure 2**) as outlined in **Exhibit B, Section 3.6** of the ISM01.1 SOW.

8.21 Software and Hardware systems are maintained on a daily basis. The network administrator, in addition to maintaining security and the integrity of all data on the main servers, also assists data technicians in the maintenance of each Windows based workstation, as outlined in **Exhibit F, Section 2.6** of the ISM01.1 SOW.

8.22 Software Updates:

Omega LIMS periodically updates the software. IT will also make periodic updates and therefore prior to using any module you should update it from the main menu.

9. Data Reduction and Calculations

NA

10. Quality Assurance/Quality Control

NA

11. Data Validation and Reporting

See **Section 8** of this SOP.

12. Corrective Action Procedures

All out-of-control situations must be corrected or qualified prior to submission of the final report. Any initial deviations should be noted in the prep/analysis notes section of the "CLP/CLP-Like Deliverable Checklist for Inorganic Analysis" so that corrections may be made or the report is returned to the laboratory for final correction.

13. Health and Safety

All employees must read and be familiar with the Mitkem Health and Safety Plan, and are required to comply with the safety practices described therein.

14. Pollution Prevention, Waste Management, and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. The Mitkem Quality Assurance Plan
2. US EPA CLP Statement of Work (SOW), for Inorganics Analysis, Multi-Media/Multi-Concentration, ILM04.1.
3. US EPA CLP Statement of Work (SOW), for Inorganics Analysis, Multi-Media/Multi-Concentration, ILM05.4.
4. US EPA CLP Statement of Work (SOW), for Inorganics Superfund Methods, ISM01.1.

Attachments:

1. **Figure 1**: CLP/CLP-Like Deliverable Checklist for Inorganic Analysis.
2. **Figure 2:** ISM01.1 DC-2 Forms

Figure 1

CLP/CLP-Like Deliverable Checklist for Inorganic Analysis

MITKEM LABORATORIES

CLP/CLP-like Deliverable Check List for Inorganic Analysis

Project Number:_____
Client._____
Input by/date:_____
Forms generated on/date:_____

Analysis:_____
Category:_____
Reviewer:_____
(1) Date Reviewed:_____
(2) Date Reviewed:_____
Corrections by:_____

Elements Required:

Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Ni	K	Se	Ag	Na	Tl	V	Zn	Sn	CN	Hg

Items:	Pages	Check	OK/Unusual Observation
Sample Log-In Sheet			
Prep Log Sheet (AQ/SL)			
% Solid Bench Sheet			
Tumbling Log (TCLP/SPLP)			

	Check	Lab ID	OK/Unusual Observation/Deviation/Flags
ICV / CCV			
Matrix Spike (N)			
Duplicate Samples (*)			
Serial Dilutions (E)			
Post Digestion Spike			
LCS			
ICP Interference			
CRA / CRI			

Prep/Analysis Notes:

	Yes	No
Client ID Check:		
ID Truncation:		
Special Request:		

Figure 2

ISM01.1 DC-2 Forms

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

MOD. REF. NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.6)

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
1.	Inventory Sheet (DC-2)				
2.	SDG Narrative				
3.	Sample Log-In Sheet (DC-1)				
4.	Traffic Report/Chain of Custody Record(s)				
5.	Cover Page				
Inorganic Analysis					
6.	Data Sheet (Form I-IN)				
7.	Initial & Continuing Calibration Verification (Form IIA-IN)				
8.	Blanks (Form III-IN)				
9.	ICP-AES Interference Check Sample (Form IVA-IN)				
10.	ICP-MS Interference Check Sample (Form IVB-IN)				
11.	Matrix Spike Sample Recovery (Form VA-IN)				
12.	Post-Digestion Spike Sample Recovery (Form VB-IN)				
13.	Duplicates (Form VI-IN)				
14.	Laboratory Control Sample (Form VII-IN)				
15.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)				
16.	Method Detection Limit (Annually) (Form IX-IN)				
17.	ICP-AES Interelement Correction Factors (Annually) (Form XA-IN)				
18.	ICP-AES Interelement Correction Factors (Annually) (Form XB-IN)				
19.	Internal Standard Association (Form XI-IN)				
20.	Preparation Log (Form XII-IN)				
21.	Analysis Run Log (Form XIII-IN)				
22.	ICP-MS Tune (Form XIV-IN)				

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	PAGE NOs.		CHECK	
	FROM	TO	LAB	REGION
23. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)				
24. Initial Calibration (Form XVI-IN)				
25. ICP-AES Raw Data				
26. ICP-MS Raw Data				
27. Mercury Raw Data				
28. Cyanide Raw Data				
29. Preparation Logs Raw Data				
30. Percent Solids Determination Log				
31. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments_____)				
Sample Tags				
Sample Log-In Sheet (Lab)				
32. Misc. Shipping/Receiving Records (list all individual records) Communication Logs				
_____				
_____				
33. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____				
_____				
34. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____				
Analysis Records _____				
PE Instructions _____				
Description _____				
35. Other Records (describe or list)				
Communication Logs				
_____				
_____				

36. Comments:

_____

_____

_____

Completed by:
(CLP Lab) _____ _____ _____
(Signature) (Print Name & Title) (Date)

Audited by:
(USEPA) _____ _____ _____
(Signature) (Print Name & Title) (Date)

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

Sample Data Control for CLP (SOM01.2)

SOP No. 110.0035

Rev. 1

	Signature	Date
QA Director:		4/8/08
Lab Director:		4/9/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Sample Data Control for CLP (SOM01.2)

SOP 110.0035 Rev. 1

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the data management procedures used in support of forms generation for CLP samples using the protocols in SOM01.2.

2. Personnel Qualifications and Responsibilities

The personnel responsible for the data management must be familiar with the protocols outlined in this SOP and follow these procedures.

3. Summary of Procedure

The CLP forms are generated using the **instructions, procedures and/or** protocols outlined in the **USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organic Analysis Multi-Media, Multi-Concentration SOM01.2 (April 2007)** and **section 8** of this SOP.

4. Sample Preservation, Containers, Handling, and Storage

N/A

5. Interferences and Potential Problems

N/A

6. Equipment and Apparatus

6.1 Intel based workstation, operating in a Windows OS.

6.2 Target Software from ThruPut.

7. Reagents

N/A

8. Procedure

HP EnviroQuant Software is used for data acquisition for all of the organic CLP analyses performed in the laboratory. The software is resident in each of the PCs (as part of a company wide network) that are password accessible Analysts are assigned different security levels depending on the job requirement.

8.1 Upon completion of each data acquisition, the raw data are copied directly to the primary file server. The data are then evaluated using Target. The Target reviewed data will then be uploaded into LIMS. The data in LIMS will need to be QA'd for forms generation (**Forms I through VIII for volatiles and semivolatile, Forms I through X for pesticides and Aroclors**) and supporting documents including chromatograms, quantitation reports, **tentatively identified compounds (TIC)** library search and spectra generation.

8.2 The laboratory owns several copies of the data processing software Target that is installed on individual workstations. The workstations are connected to a file server via the company's local area network (LAN). For more details, see SOP No. 110.0029, Electronic Data Management.

8.3 Target data and methods reside on a system with name "Organics". The system is configured as follows:

8.3.1 Volatiles analysis:

Organic/VOA/Vi/Batch/File, where i = 1,2,5,or 6.

8.3.2 Semivolatile Analysis:

Organic/SVOA/Sj/Batch/File, where j = 1,2,3 or 4

8.3.3 Pesticide and/or Aroclor Analysis:

Organic/SVOA/Ek/Batch/File, where k = 1,2, 3, 4 or 5.

Where Vi is the instrument that is used in the volatile lab; Sj is the instrument that is used in the semivolatile lab; and Ek is the instrument that is used in the Pesticide/PCB lab.
Batch is the batch of runs that are generated within the analytical batch. This is typically the date the mass spectrometric analysis is acquired. For pesticides/PCB, the batch is the date the analytical sequence was initiated.

Under the subdirectory **File** are the raw data files that are uploaded from the Hewlett Packard EnviroQuant Software. This subdirectory also contains the various method files that are used to process data including tunes, initial calibration, continuing calibration and sample analysis for mass spectral based analysis, and initial calibration, calibration verification and sample analysis for GC based analysis. These method files are as follows:

8.3.4 Volatiles (Trace and Low/Medium)Analysis:

Vi_BFB_SOM.m for evaluating BFB tunes for Vi.
Vi_TVOA_1.2.m for evaluating analysis of blanks and samples by trace volatiles or Vi_SOM1.2_LS for low/medium volatiles, where i = 1,2,5, or 6.

8.3.5 Semivolatile Analysis:

Sj_dftppSOM.m for evaluating DFTPP tunes for Sj.
Sj_SOM.m for evaluating analysis of blanks and samples, where j = 1,2, or 3.

8.3.6 Pesticide/PCB Analysis:

Ek_somPEST-F.m for evaluating sample analysis (including all standards) for Ek front column;Ek_somPEST-R.m for evaluating sample analysis (including all standards) for the rear column, where k = 1,2, 3, 4, or 5.

Once the data are processed, the analyst will evaluate the data to ascertain that all of the peaks of interest are properly integrated. If needed, manual integration will be performed to improve data quality. The analyst who performs the manual integration will initialize and date all such changes. In addition, hardcopy of the changes are generated as part of the audit trail. Most of the time, hardcopies of the re-integrations are self-evident and the analyst does not need to further justify the re-integrations. The lab supervisor or his/her designee will review and sign off the changes. See Mitkem SOP # 110.0008.

8.4 Procedures for Setting up Mass Spectral Based Analysis:

8.4.1 Mass spectral based analyses including the volatile and semivolatile organic analyses are similar and will be discussed together.

8.4.2 As discussed earlier, the various mass spectral processing methods are provided by the vendor and modified by the laboratory. On the day the samples are **to be** analyzed, the method file is created by going into Target/Edit/Copy to copy the methods from the previous batch of samples that were processed using the SOM01.2 approach.

8.4.3 **Make sure that both the tune and sample analysis methods are copied.** After the methods are copied, rename the methods to the date the actual batch of data are to be processed.

8.4.4 Use Target/Process/Select Method to select the tune method.
Use Target/Process/Process to process the tune analysis.
Use Target/Reports/Review Reports/Target Graphics to evaluate the tune analysis Whenever the ion abundance is outside the required criteria, it will be flagged with a qualifier "*". The analyst should re-analyze the tune sample. If the failure to meet tune criteria persists, the analyst should identify and resolve the problem by following the procedures outlined in the analytical SOP.

8.4.5 After the tune analysis passes criteria, proceed to the calibration standard analysis. Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the initial or continuing calibration standard(s). Use Target/Review/Target Review to evaluate the target analytes in the analysis for the initial or continuing calibration; use manual edition if needed.

8.4.6 For initial calibration, use Target/Process/Edit to open the method-editing window. Use View/Initial Calibration to evaluate the RRF and %RSD for each target analyte. An arrow qualifier will flag the target analyte(s) that does not meet criteria. Only up to two compounds may be flagged, and these compounds must have a RRF no less than 0.01 and a %RSD not greater than 40.0%. Corrections as noted in the analytical SOP should be taken to meet these criteria by re-analyzing one or more levels of the initial calibration.

8.4.7 For the continuing calibration, use Target/Reports/Review Reports/Target Sample to evaluate the RRF and %D for each target analyte. An arrow qualifier will flag the target analyte(s) that does not meet criteria. Only up to two compounds may be flagged, and these compounds must have a RRF no less than 0.01 and %D not greater than 40.0%. Corrections as noted in the analytical SOP should be taken to meet these criteria by re-analyzing the calibration standard. Once the daily calibration is processed, the RRF will automatically be updated in the method file. The same method file is then used to process the method blank, LCS, and samples run within the batch.

8.4.8 Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the method blank, LCS, and all samples. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and all samples; use manual edition if needed. Use Target/Reports/Review Reports/ISTD Monitor to evaluate the internal standard recovery. Use Target/Reports/Review Reports/Recovery Report to evaluate the recoveries for the system monitoring or surrogate compounds. For the LCS, the recoveries for the spiked compounds are listed as well. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and

samples. Some corrections as noted in the analytical SOP may be needed if the QC samples do not meet criteria.

8.4.9 After all the samples within the SDG, which may include several batches of samples, have been processed and reviewed, use Target/Reports/QuickForms to generate the required forms. The analyst should include all the files, which include all samples, tune, initial calibration, continuing calibration, method blank, LCS, and other necessary QC samples for the SDG using Add and Remove buttons.

8.4.10 Under the QuickForms Host: enter the case number and lab contract number into the appropriate boxes. Use Edit/Forms Control and then select SOM under Form Set. Make sure Fraction Type is set as VOA for volatile analysis or SV for semivolatile analysis; the analyst has the option of generation of all the required forms (**Forms I through VIII**) at once or one at a time. Use Edit/Edit SDG to evaluate all the files in the SDG. All the samples analyzed in the same batch will be organized into one QC group with their associated tune, initial calibration standard or continuing standards, and QC samples. Use Add Sample, Delete Sample, Delete QC Group, and New QC Group to rearrange samples or QC groups if needed. Use Edit/Blank Test to associate sample contamination with the corresponding method blanks. This step must be performed before producing any Form **I** to ensure that the compound has been flagged with a "B" qualifier if the same compound is also detected in the associated method blank. Use Generate/Generate Reports to produce all the form as selected. Use Generate/Review Reports to evaluate the forms.

8.5 Procedures for Setting Up Pesticides and/or Aroclor Analysis:

8.5.1 As discussed earlier, the Pesticides/PCB processing methods are provided by the vendor and modified by the laboratory. On the day the samples are to be analyzed, the method file is created by going into Target/Edit/Copy to copy the methods from the previous batch of samples that were processed using the SOM01.2 approach. **Make sure that both methods for the front and rear columns are copied.** After the methods are copied, rename the methods to the date the actual batch of data are to be processed.

8.5.2 Only the front column is discussed below, since the procedures are the same for the rear column.

8.5.3 Use Target/Process/Select Method to select the front column method. Use Target/Process/Process to process the initial calibration in the same order as the standards are analyzed, i.e. starting with the resolution check and ending with the INDABH. Use Target/Review/Target Review to evaluate the standard analyses to make sure they conform to method specifications as discussed in the Pesticide Analysis SOP. Some corrections, such as re-analysis of some of the levels of the calibration standards, may be needed to complete the initial calibration, as the method requires. Use manual edition if needed. Use Target/Process/Edit to open the method-

editing window, then use View/Initial Calibration to evaluate the %RSD for each target analyte in the initial calibration.

8.5.4 Once the initial calibration is processed, the CF will automatically be updated in the method file. The same method file is then used to process the method blanks, LCS, continuing calibration, and all samples run within the batch. Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the method blank, LCS, and all samples. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and all the samples; use manual edition if needed. Use Target/Reports/Review Reports/Target Sample to evaluate the CF and %D for each target analyte for the continuing calibration standard. Use Target/Reports/Review Reports/Recovery Report to evaluate the recoveries for the system monitoring compounds. For the LCS, the recoveries for the spiked compounds are listed as well.

8.5.5 After all of the samples within the SDG, which may include several batches of samples, have been processed and reviewed, use Target/Reports/Quick Forms to generate the required forms. The analyst should select all the files, that includes all samples, Resolution Check, PEM, instrument blank, initial calibration, continuing calibration, method blank, LCS, and other necessary QC samples for the SDG using Add and Remove buttons.

8.5.6 To generate Organics CLP forms, refer to the 'Organics Forms Reporting' document found in General Mitkem Omega Information on the Mitkem switchboard (http://switchboard.mitkem/docs.asp).

8.6 Data Package Assembly:

8.6.1 Once all of the required forms are generated; also generate all of the supporting documents. These include chromatograms and quantitation reports for the tune, initial calibration, continuing calibration, samples, blanks, LCS, TIC library search and spectra of positive hits for the mass spectral based analysis. For pesticides analysis, these include chromatograms, manual integration hard copies and quantitation reports for all initial calibration, calibration verification, florisil check, samples, blanks, LCS. All the information and documentation is forwarded to the document control officer with the following:

a. Copy of the extraction prep log.
b. Tracking log from sample receipt
c. Extraction transfer log.
d. Instrument run log.

The document control officer will assemble the data package per the SOW instructions.

8.7 Software Updates:

8.7.1 ThruPut is no longer updating the Target Software.

8.7.2 At Mitkem, the software and hardware systems are maintained on a daily basis. The laboratory has a full-time network specialist. This person is also responsible for monitoring the automatic copy/backup of all data each night.

8.7.3 At Mitkem, data base integrity (e.g. quantitation list, initial calibration and continuing calibration updates) is maintained by the analyst and his/her lab supervisor. Changes, if made, are reviewed by another analyst and/or supervisor.

9. Data Reduction and Calculations

N/A

10. Quality Assurance/Quality Control

N/A

11. Data Validation and Reporting

N/A

12. Corrective Action Procedures

All out of control situations must be recorded in the corrective action logbook and documented returned to control as described in SOP No. 80.0007.

13. Health and Safety

All employees should read and be familiar with Mitkem's Safety Manual and Chemical Hygiene Plan, and comply with the safety practices described therein.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories.

USEPA. Statement of Work, Method SOM01.2.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

SOP No. 110.0038

Rev. 7

	Signature	Date
QA Director:		2/19/09
Lab Director:		2/19/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

Rev. 7

1. Scope and Application

This Standard Operating Procedure (SOP) describes the procedure for measuring the percent solids/percent moisture of many types of solid material. An undried portion of sample is weighed and dried at 105 ° C. The sample is cooled, reweighed and the percent of water evaporated provides the result.

Note: It may be necessary for some specifically identified samples to undergo a pre-drying process. See amendment.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing their work tasks following the procedures outlined in the SOP. Supervisors and managers are responsible for reviewing and approving new and revised SOPs. They are responsible for ensuring that SOPs are accurate and up to date, and are being implemented appropriately.

3. Summary of Procedure

Any standing water in the sample container is decanted. Samples are mixed to ensure sample homogeneity. Percent solid is calculated by the evaporation of water in a solid sample to a constant weight in a 103-105 ° C oven. .

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are stored prior to percent solids determination at a temperature between 2-6° C.

4.2 There is no "official" sample holding time. This analysis should be performed as soon as possible after sample receipt.

5. Interferences and Potential Problems

5.1 Temperature fluctuations will cause measurement errors.

5.2 Balance fluctuations can cause measurement errors. Also, if the balance is not properly calibrated, errors can occur.

5.3 Negligence in taring the balance prior to recording weights will also cause errors.

5.4 Results of less than 50% solids for ISM01.1 soils from EPA require immediate SMO notification and guidance.

5.5 If a SOM01.2 soil sample has greater than 65% moisture, the laboratory may use up to 50 g of soil sample in order to achieve the expected CRQLs. The amount of sample used and the % moisture should be noted in the SDG Narrative

6. Equipment and Apparatus

6.1 Aluminum weighing pans.

6.2 Top Loading balance, capable of reading to two decimal places, connected to a PC loaded with the Balance Talk software program.

6.3 PMOIST Binder to store photocopies of the Balance Talk worksheets

6.4 Scoopulas for portioning the sample.

6.5 Metal pans for moving samples in and out of the oven.

6.6 Class "S" or Class "2" weights for balance calibration.

6.7 Drying oven capable of maintaining 103-105 ° C (ISM01.1 allows 105 ± 5° C).

6.8 Class "1" weights for monthly calibration/verification .

6.9 Dessicator.

7. Reagents

Not applicable.

8. Procedure

Sample Prep: (***It maybe necessary to "pre-dry" some samples. See amendment, sec. 16.)***

8.1 Run the PMOIST backlong in LIMS by due date. Select the samples for PMOIST.

8.2 In LIMS, go to the Data Entry page. In the data entry page, click on **Export to Excel**. In the **Test Code** field, select PMOIST.

8.3 Remove the check marks from the **MBLK** and **LCS,** and select the **Excel** button. **SAVE** the file as a temporary excel file and select the **OPEN** button. When prompted for confirmation, select the **YES** button. The temporary excel file should be named *aaayymmddx*: where aaa are the initials of the person performing the PMOIST, yy is the last two digits of the year, mm is the month, dd is the day and x is A, B, C, etc., which is the sequence number. For example, the second temporary spreadsheet on January 14, 2009 would be called arh090114B.

8.4 In the temporary spreadsheet, delete any samples in which PMOIST will not be performed yet.

8.5 Add any sample DUPs now. Sample DUPs are performed every 20 samples. To add a sample DUP, highlight the row with the sample that a duplicate analysis is to be performed upon. Copy the highlighted row and select Insert Copied Cells option. Add the suffix DUP to the end of the ID for the sample that was just copied.. Be sure to save the changes made.

8.5.1 Duplicate samples for ILM05.4 projects must be done on the same sample that is chosen for Matrix Spike. The Duplicate analysis result is actually reported on Form 6 of the data package.

8.6 Label the aluminum weighing dishes with the Mitkem sample ID .

8.7 Minimize the Omega screen and the saved temporary spreadsheet. On the desktop, double click on the My Network Places icon. Double click on Public in Avogadro, Lims and then Pmoist. Scroll down and select file newestPmoist. If a macro warning box is displayed, choose the button **Enable Macros**.

8.8 Using the **Save As** option in the File Menu save the opened workbook using the following notation: YY =last two digits of the year; MM = numeric month example (June = 06, July = 07) DD = numeric value of the date of the month, X = letter corresponding to sequence number for the day. Do not forget to save this file to the Avogadro/ Public/Lims/**PMoist** directory.

8.9 Move to the **Header Information** page. Select the **Clear Data** button to remove any old data remaining from a previous batch. Complete the **Date and Time In/Out** and **Temp**erature fields, and save to the new **PMoist** workbook.

8.10 Copy Mitkem Sample IDs from the temporary Excel spreadsheet starting with cell A2 to D2 down to Ax and Dx, where x is the number of the last row of samples.

8.11 Paste Mitkem Sample IDs into the Sample field starting in cell (A2) of the Data Entry page of the new **PMoist** spreadsheet. The temporary spreadsheet should be closed at this point.

8.12 The balance calibration must be checked and recorded in the appropriate logbook. Use calibration class "S" check weights of 1.0, 10.0 and 100 grams. If the balance does not meet the calibration check criteria listed in the logbook, do not proceed, but inform the supervisor.

8.13 In the spreadsheet move the cursor to cell H2 (first cell under Tare Weight column). Begin to collect the weight of the weighing tin: make sure the balance is zeroed. Place a weighing tin on the balance. When the balance displays the "*" press the ''print'' button on the balance. The tare weight is then printed in the highlighted cell of the spreadsheet. The cursor automatically goes to the next cell (Tare + Sample).

8.14 Open the sample jar. If there is any free water standing on the surface of the sample, this is carefully decanted (poured) into a waste container. Using a clean scoopula, the sample in the jar is mixed to insure it is homogeneous (well mixed).

8.15 Collect the weight of the wet sample and weighing tin. Measure out approximately 5-10 grams of sample. The weights are recorded to the nearest 0.01 gram. When the balance displays the "*" press the print button on the balance.

The weight of the weighing tin and sample in then printed in the highlighted cell (Tare + Sample). The cursor automatically goes to the next Tare Weight cell

8.16 Proceed with **steps 8.13 to 8.15** until all samples are done. The spreadsheet should be saved periodically.

8.17 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.

8.18 As each sample is weighed, its tin is placed on a metal tray. As the tray of samples is complete, it is carefully placed on a shelf in the 103-105 ° C drying oven. Samples are dried overnight, optimally for at least 12 hours. ILM5.4/ISM01.1 soils have a 12 hour minimum drying time, and a 24 hour maximum.

8.19 If for some reason the samples cannot be dried overnight, a shorter drying time may be used, provided the sample is dried to a demonstrated constant weight. This procedure can only be used with the Supervisor's approval and must be carefully documented. The weighed wet sample is placed in the drying oven for at least four hours. Following four hours, the sample may be removed from the oven, cooled in the dessicator, weighed, and returned to the oven. After an additional 30 minutes of drying in the oven, the sample may be removed, cooled, reweighed, and replaced in the drying oven for an additional 30 minutes of drying. If two consecutive weights are equal (within 4%) then the sample has been dried to a constant weight, and the percent solids may be calculated.

This procedure must be documented in the PMOIST binder. A note is made for the sample that undergoes this procedure. The reverse side of the page is used to record the initial wet sample weight and time sample is first placed in the oven. When the sample is removed for weighing, the time and weight are also recorded on this page. There must be at least three weights and times recorded (initial and three consecutive constant weights).

ILM5.4/ISM01.1 samples which cannot be dried for the full 12 hours must also have demonstration of a constant final weight. Data must be recorded for a minimum of two repetitive 'weigh/dry/desiccate/weigh' cycles with a minimum of 1-hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

8.20 DAY TWO: Open the saved **PMoist** spreadsheet from the previous day. Enter date and time the samples were taken out of the oven and the oven temperature. Place the cursor so that it is in cell J2 (Final Wt). Ensure the balance has been calibrated and is zeroed as described above.

8.21 The tray of samples is carefully removed from the oven and the samples are placed in a dessicator to cool.

8.22 Put the cooled, dried sample and pan on balance. When the balance stabilizes press the **Print** button on the balance.

8.23 Select the **Calculate Pmoi**st button. Click again on the Data Entry sheet of the worksheet if the calculations appear acceptable. On the header information page select the **Copy Data** button. Click on the **Print Report** button. Save and close the Excel program.

8.24 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.

8.25 From the main page of Omega click on the **Data Entry** button.

8.26 Select the **Add** button. From the instrument ID dropdown box select Dry Weight Oven (DWO). Complete the Run Start Date field. From the Analyst field dropdown box select the analyst name. Select the **Data Import** button.

8.27 From the **Specification** dropdown box select **Omega Excel File. S**elect the **Run Import** button. From the **Open Dialog** box select the **PMoist** file and then select the **Open** button. Leave the value of 1 in the Text box and then select the **OK** button in the Spreadsheet Range box.

8.28 After Omega finishes importing the data, select the Data button. Of the three fields only the raw field should have value. Select the **Calc Seq** button and then select **Yes** when asked to continue.

9. Data Reduction and Calculations

The software performs all calculations. The results are reviewed by an independent analyst and then verified in the LIMS Omega System.

10. Quality Assurance/Quality Control

10.1. A duplicate sample must be performed at least every 20 samples.

10.2. The balance calibration must be within specifications.

10.3. The balance must be calibrated monthly with the Class "1" weights located in the QA/QC Office.

10.4. The drying oven temperature must be stable in the range of 103-105° C.

10.5. Times samples are put into and taken out of the oven are recorded.

11. Data Validation and Reporting

Results are checked first by the analyst and then by the supervisor. The print-out of the completed Excel spreadsheet is delivered to the Supervisor or designee for review.

Following Supervisor review and approval, all data are QA Validated in the Omega LIMS. The Excel spreadsheet is dated and sequentially numbered and scanned into the PMOIST optical file database for permanent storage. The original hardcopy record is saved in the PMOIST binder.

12. Corrective Action Procedures

12.1 If the balance calibration exceeds acceptance criteria, recalibrate it according to manufacturer's instructions.

12.2 If the drying oven shuts down during the night, the analysis will need to be restarted, but samples will not need to be re-weighed.

12.3 If drying oven temperature exceeds 110° C, samples will need to be reprepped and reanalyzed.

12.4 If a sample spills during the final weighing, it will need to be reprepped.

12.5 If a sample and duplicate RPD vary more than 35%, another independent analysis of the sample must be performed.

12.6 If sample drying time is not within 12 to 24 hours for ILM/ISM soils, the time must be noted for inclusion in the project narrative.

13. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of samples used in the method. Lab coat, gloves and safety glasses must be worn at all times in the lab.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

APHA, AWWA, and WEF, "Standard Methods for the Examination of Water and Wastewater," 18th edition.

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Organic Analyses (Multi-Media, Multi-Concentration) SOM01.2

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Analyses (Multi-Media, Multi-Concentration) ILM05.4

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM01.1

Balance Talk user manual

16. Amendment to SOP

16.1 Due to some sample matrixes and/or special client requests, it may be necessary to "pre-dry" samples prior to the sampling procedure. These samples will be specifically identified by the Laboratory Supervisor or Project Manager.

16.2 Visually determine or weigh out approximately 15 – 20 grams of each sample in an appropriate container. (An aluminum pan should be sufficient)

16.2.1 Place each sample in a drying oven set at 60°C overnight.

16.2.2 Follow the procedure as described in **Section 8**.

APPENDIX E

- MEETING MINUTES FOR MARCH 16, 2009 PARTNERING MEETING
- MEETING MINUTES FOR MAY 14, 2009 PARTNERING MEETING
- MEETING MINUTES FOR JUNE 17, 2009 CONFERENCE CALL

MEETING MINUTES FOR MARCH 16, 2009 PARTNERING MEETING

Meeting Minutes
March 16, 2009 Meeting in Boston, MA
NSB-NLON, Groton, CT

Meeting Location: EPA Region I, Boston, MA

Agenda Topics:

1. Non-Time Critical Removal Action Work Plan for sediment at Pier 1 Inner and Outer Areas (Response to EPA comment letter dated 20 January 2009).

2. Follow-up issues from January 28, 2009 comment resolution meeting on the Draft Lower Subase Feasibility Study.

3. Area A Wetland Remedial Investigation.

Handouts for all three topics were provided prior to the meeting by email and passed out at the meeting.

Minutes Recorded by: N. Balsamo

Participants:

U.S. Navy (Navy): Dick Conant, Val Jurka

Tetra Tech NUS (TtNUS): Corey Rich, Nina Balsamo, Aaron Bernhardt, Jean-Luc Glorieux (by phone)

U.S. Environmental Protection Agency (EPA) Region I: Kymberlee Keckler, Bart Hoskins, Dave Peterson, Chau Vu

Gannett Fleming (GF): Greg Kemp, Todd Finlayson

Connecticut Department of Environmental Protection (CTDEP): Mark Lewis, Rosemary Gatter-Evarts (by phone)

Tetra Tech EC (TtEC): Dan Sullivan (by phone, Topic 1), Roxanne Clarke (Topic 1)

U. S. Fish and Wildlife Service (USFWS): Ken Munney

National Oceanic and Atmospheric Administration (NOAA): Ken Finkelstein (Topic 1)

Time of Meeting: 9 AM to 5 PM

Risk: Prior to addressing Topic 1 on the agenda, risk issues were discussed with C. Vu (EPA).

Topic 2 – Lower Subase Feasibility Study

Oak Ridge – C. Vu stated that Region 9 had stopped updating PRG tables in 2004, but said that Oak Ridge values were applicable for all media. EPA Region 1 has been recommending that the Oak Ridge values be used in risk assessments instead of Region 9 values. It is unclear whether risk will increase or decrease using the Oak Ridge values. Tetra Tech will revise the human health risk assessment using Oak Ridge 2008 values.

TCE – Tetra Tech used EPA draft TCE toxicity values for the Draft Lower Subase FS; Tetra Tech suggested California EPA numbers for the revised risk assessment for the Draft Final FS. C. Vu agrees with the California EPA cancer slope factor and inhalation unit risk number, but suggests

the NYS DOH non-carcinogenic Tier 3 value (10 $\mu g/m^3$). Tetra Tech will to revise the risk assessment to use the California (carcinogenic) and NYS DOH (non-carcinogenic) values.

Topic 3 – Area A Wetland Remedial Investigation

A. Bernhardt commented that for surface and groundwater data, he will use the maximum values from all rounds for initial screenings for the human health risk assessment. C. Vu agreed and said the EPA asks that the maximum concentrations be used for all media for the initial screening.

Topic 1: Non-Time Critical Removal Action Work Plan for sediment at Pier 1 Inner and Outer Areas

Pier 1:

D. Conant passed out copies of the January 20, 2009 comment letter from K. Keckler to Nina Johnson (Navy) that was amended to include Navy responses. In the response to comment GC1, 1st paragraph, the Navy suggested additional Inner Pier 1 sampling at three locations and two depths (surface and deep) per location, for a total of six additional samples. The samples will be analyzed for PCB Aroclors to address TSCA issues. The EPA agreed that six samples were reasonable but that some samples could be taken at other locations if the sediment thickness did not allow six samples to be taken at the proposed locations. The Navy asked what was needed regarding a sampling and analysis plan (SAP). The EPA stated that the SAP prepared for the sediment sampling for the Lower Subase Feasibility Study could be amended.

The Team discussed control of turbidity during Inner Pier 1 dredging. The Navy proposed monitoring turbidity for 1 week prior to the start of dredging to get a baseline value; weather, tide and wind will be noted. During dredging operations, turbidity will be monitored continuously outside the turbidity curtain with one monitor near the water surface and one monitor 1 to 2 feet above the river bottom. It was discussed which would be better - a double turbidity curtain or another monitor. D. Sullivan stated that a second curtain would be cumbersome and unnecessary since the turbidity curtain would remain closed when excess turbidity was present. During dredging Inner Pier 1 dredged sediment will be placed in a 600-700 cubic yard barge and one barge per day will be filled at the proposed dredging rate. The Navy stated that if turbidity passed through the curtain, work would stop and the curtain would be reconfigured. R. Clarke said the monitor could be set with an alarm for a specific reading, such as 20% above the baseline value. G. Kemp suggested that the Work Plan include how to open the curtain and how to move the monitor out of the way of the barge. Visual observation and manual portable meters were discussed. The Navy requested that TtEC look into measuring turbidity inside the curtain prior to opening the curtain. Other issues to be addressed include whether the tug can go inside the curtain, and preventing "prop wash" from the tug boat propeller in the sediment. The team also discussed how long it would take to allow sediments to settle out prior to opening the silt curtain. It was suggested that letting the area settle overnight should be adequate for the fines to settle out and allow the silt curtain to be opened.

Conclusion: The Inner Pier 1 Work Plan will be revised to more clearly address turbidity monitoring and operational procedures, such as when the turbidity curtain can be opened, preventing "prop wash", and coordination of the tug, barge, and turbidity curtain.

Comment GC3: D. Sullivan explained that an environmental clamshell would not be practical in Inner Pier 1 because there would be >50% liquid in shallow environments and environmental clamshells are not good in corners. It would be better to open a smooth-edge excavation bucket and raise it slowly.

The sequence of expected Pier 1 events was discussed. Pier 1 is expected to be removed in April/May of 2009. First the deck will be removed, and then the wooden piles will be pulled. Piles that don't snap off at the mudline will be cut at the mudline by a diver with a hydraulic chainsaw.

Figures for the Topic 2 - Item 4 discussion showing the sediment concentration contours were used to discuss the possible footprint of the proposed Outer Pier 1 excavation. It was mentioned that some ERM-Q values had changed. It was discussed how to coordinate dredging Inner Pier 1 and Outer Pier 1 sediment. TtEC estimated 5 weeks to dredge Inner Pier 1. EPA expressed a preference for dredging Inner Pier 1 and Outer Pier 1 sediment at the same time, but the footprint of Outer Pier 1 sediment has not been determined and the current budget of $2.1 million for Inner Pier 1 will likely not be adequate for both Inner and Outer Pier 1 sediment dredging. K. Keckler said that any Outer Pier 1 sediment exceeding criteria that is not removed during the Inner Pier 1 dredging will be handled under the Lower Subase FS.

The Navy asked if they remove sediment exceeding criteria to 6 feet and confirmation sampling indicates the remaining sediment exceeds criteria, what do they need to do? EPA stated that if either criteria (Total ERM-Q>1.17 or PCB> 1 ppm) were exceeded, they need to keep going. If the chemical concentrations in the surface sediment do not exceed criteria, but the concentrations in the subsurface sediment do exceed criteria, land use controls may be necessary.

Summary of Pier 1 discussion:

- Sampling Inner Pier 1 sediment for PCBs at three locations/ six samples is acceptable to EPA.
- Operational procedures related to turbidity (curtain/metering/tug/barge) will be more clearly identified.
- The extent of proposed Outer Pier 1 dredging is not fully determined.
- The Navy will look into possible additional funding to apply to dredging Outer Pier 1 sediment during Inner Pier 1 dredging.

Zone 4:

Figures for the Topic 2 - Item 4 discussion were used to discuss the limit of dredging sediment from the Thames River adjacent to Zone 4. D. Conant stated that the Navy's proposed maintenance dredging line will be from sample SB-4 to SB-3, continuing straight to the edge of the maintenance dredging area (shown as orange on figures), plus a 30-foot buffer to the west of the maintenance line. The buffer zone will be sloped at 3 horizontal to 1 vertical from the CERCLA sediment to the maintenance dredging area. Maintenance-dredged sediment will go to the CAD cell. D. Conant stated that they are targeting September/October for maintenance dredging. Submarines are not berthed at Zone 4; therefore, maintenance dredging is not needed beyond the maintenance line. Sediment at SD-007 from 0-1 ft and SD-008 may be dredged during maintenance dredging.

C. Rich stated that between the maintenance and CERCLA dredging actions, the sediment at SD-008 will be remediated. C. Rich stated that all of the PCB exceedance locations were within the CERCLA dredging area. CERCLA dredging will include the area east of the CERCLA line in the Pier 6 dredging area, designated as blue cross hatch on the figures.

EPA requested that the Navy maintenance dredging/buffer zone/CERCLA lines be shown on the drawings. The CERCLA dredging limit will be determined during the Lower Subase FS.

The stability of the quay wall was briefly discussed. Based on previous discussions with NSB-NLON Public Works, for the purpose of the Lower Subase FS, it will be assumed the quay wall will be stable if the top 5 feet of adjacent sediment is removed and immediately replaced with clean material. Further evaluation of quay wall stability during remediation will be included in the final design.

Action Items:

- The Navy will look into possible additional funding to apply to dredging Outer Pier 1 sediment during Inner Pier 1 dredging.
- D. Conant will send M. Lewis and K. Keckler blind copies of the cover letter of the Corps of Engineers application for the maintenance dredging.
- C. Rich to send out revised ERM-Q tables and how ERM-Qs were calculated for both Pier 1 and Zone 4 areas.
- C. Rich to provide EPA with a description of how the ERM-Q contour lines were generated on the sediment maps for both Pier 1 and Zone 4 areas.

Topic 2: Follow-up Issues from January 28, 2009 Comment Resolution Meeting on Draft Lower Subase FS (Remaining outstanding issues)

Item 1. Copper in Zone 1 groundwater and CTDEP approval of 100 dilution factor for Zone 1.

C. Rich reviewed the handout for Topic 2 - Item 1. As discussed in the January 28 meeting, copper was reported in Zone 1 at the power plant intake and outfall. The reason for reported copper results groundwater/surface water/ power plant has not been established.

C. Rich reviewed historic groundwater data in the handout and noted that turbidity and salinity may have caused interference and higher metals concentrations in some samples. R. Gatter-Evarts reiterated that the SWPC is based on total analyses, not filtered. Sample locations are listed in the tables. Tetra Tech's conclusion is that copper migration is not a concern based on average concentrations, lack of trends from several rounds of sampling, coupled with distance from river analyses . D. Conant mentioned that the draft report on the cooling water should be out in about a month and may have more information relative to interference.

Tetra Tech asked whether CTDEP agreed with the copper evaluation. Does this approach summarize the concerns? Is the issue resolved without additional sampling?

CT_DEP had not reviewed the data package and a March 25th follow-up call was to be set-up to discuss.

Action Item:

- M. Lewis and R. Gatter-Evarts to review the Topic 2 - Item 1 handout and send their resolution to the Team by email.

Item 2. Review resolution status of issues related to alternative SWPC and PMCs and RCRA Hazardous Waste Regulations/CTDEP RSRs.

C. Rich reviewed the handout for Topic 2 - Item 2. C. Rich reviewed the Alternative PMC items on which the CTDEP had issued approvals since the January 28 meeting, including LNAPL, infiltration, and background groundwater.

Action Item:

- Mark Lewis to provide written approval of the infiltration calculations by email.

Attachment 1 was reviewed by C. Rich and J. Glorieux. The Attachment 1 memo and tables present and evaluate arsenic groundwater data in all seven zones compared to an Alternative SWPC of 10 µg/L. In Zone 1, higher arsenic concentrations were found in the same round and wells that had the higher copper concentrations, indicating the same interference issues relating to turbidity and salinity. Reported copper results were low at those same wells as arsenic in the Lower Subase RI samples.

Based on the information provided in Attachment 1, Tetra Tech is looking for concurrence from CTDEP that arsenic should not be retained as a groundwater COC for the Lower Subase FS.

Action Item:

- R. Gatter-Evarts to review Attachment 1 of the Topic 2 - Item 2 handout.
- C. Rich to set up a conference call with M. Lewis and R. Gatter-Evarts for 9 AM on Wednesday, March 25 to discuss arsenic and copper issues.
- CTDEP 02/18/09 email provided approval of 23 µg/L Alternative SWPC for phenanthrene. Navy to submit a request to CTDEP to formally approve this Alternative SWPC.

C. Rich reviewed the Topic 2 – Item 2 handout regarding RCRA Hazardous Waste Regulations/CTDEP RSRs, including information on the Raymark site taken from the EPA website. Raymark is an EPA-lead site in Connecticut. The PMC and "20 times rule-of-thumb" for lead were not used at the Raymark site.

D. Peterson said that the Raymark site is a unique case, unlike any other case and not relevant to this site or any other site. The "Raymark mix" was not how EPA typically defines waste; it was based on a settlement because EPA needed to define for what Raymark was liable. D. Peterson stated that where there are no TCLP tests in the Lower Subase, we need to use the 20 times rule of thumb using mass lead results. C. Rich stated that we don't have to follow CERCLA if we don't have risk. D. Peterson stated that within areas defined by the risk/cleanup goal (such as 400 mg/kg for residential lead), if levels exceed TCLP or the 20 times rule-of-thumb regarding toxicity, then RCRA standards are relevant. C. Rich commented that the State is not concerned with PMC exceedances below the groundwater table. D. Peterson stated that high lead concentrations in saturated soil are not under Connecticut jurisdiction, but as a hazardous waste issue we would still need institutional controls or a soil management plan to prevent digging.

D. Peterson mentioned that if any sources are post-1980 (not expected to apply to New London Lower Subase), they will be held to different cleanup standards (RCRA). D. Conant indicated that none of the sources are likely to be from post-1980.

C. Rich referred to Attachment 2 and requested EPA's input on streamlining the Lower Subase FS ARARs tables similar to Raymark. D. Peterson wants tables that the public can use to compare alternatives. K. Keckler asked what changes we can make to the tables to meet that objective.

Action Item:

- EPA to get back to the Team on the issue of reducing the number of ARARs tables.

Item 3. PDI "strawman" for Zone 7 lead.

N. Balsamo reviewed the general Zone 7 information on the Topic 2 - Item 3 handout. N. Balsamo passed out copies of Tables 10-1 and 10-2 from the Lower Subase RI showing Zone 7 sampling and analysis to date.

EPA had presented suggestions for a Zone 7 pre-design investigation (PDI) in an email dated October 8, 2008. N. Balsamo presented the EPA-suggested sampling locations on a 24"x36" map of Zone 7 and EPA-suggested analyses on a table. The EPA-suggested PDI included a minimum of 67 samples at 37 locations, including two rows of 10 samples west and one row of 10 samples east of Building 456.

K. Keckler stated that the two rows of 10 samples west of Building 456 appeared excessive. D. Conant stated that east of Building 456 access for sampling equipment was poor, as this area is a railroad embankment on a 45 degree slope. V. Jurka suggested doing XRF east of Building 456. G. Kemp stated that XRF can be fairly accurate, depending on what you get.

N. Balsamo described an alternative approach to planning the PDI using geostatistics. Sample locations would be determined using a two-step approach. A three-dimensional kriging map could be determined using existing data, and then the local uncertainly model could be used to determine a three-dimensional uncertainty map, which could be sliced at various depths. C. Rich thought that for the EPA suggested method, based on "biased sampling", that the sample density was too high, and suggested doing statistics to alleviative uncertainty, then filling data gaps. K. Keckler said the EPA has approved both approaches. K. Keckler suggested calling Steve Parker in Tetra Tech's Boston, MA office to see what was done at Newport. D. Conant mentioned that we need a method that is exportable to the other zones.

V. Jurka suggested consideration of XRF. B. Hoskins stated that the sample cups used to conduct XRF analysis can be retained and used to submit a sample to a fixed lab for confirmatory analysis of the actual XRF samples. V. Jurka suggested confirmatory analysis on 10% of field XRF samples.

C. Rich stated that the PDI could be done prior to the Lower Subase FS to better define the extent of contamination, or the FS can use the current data set showing conservative (large) volume and cost estimates, with the PDI done afterward to determine the exact extent. The Navy will decide later when to do the PDI.

Action Items:

- Tetra Tech to carry forward the geostatistic method to determine the suggested sampling locations for the Zone 7 PDI. It is hoped that the evaluation can be done and presented at the next meeting to be scheduled for the end of April.

- A meeting will be scheduled with the EPA for the end of April to review the results of the geostatistical PDI approach and compare to the EPA-suggested sampling and analyses.

Item 4. Thames River sediment cleanup goals and establishment of "CERCLA Line" in Zone 4 and Pier 1.

This topic was discussed during the Topic 1 discussion and was not discussed further.

Topic 3:

Item 1: Area A Wetland Remedial Investigation.

A. Bernhardt (TtNUS) reviewed the handout for Topic 3 - Item 1 and passed out maps showing sample results for copper, lead, zinc, total aroclors, total DDT, total PAHs, average PEC-Qs (metals), average PEC-Qs (with DDE), and average PEC-Qs (with DDX). Rationale and acceptability of results based on comparison to two references were discussed between A. Bernhardt, K. Munney, and B. Hoskins. B. Hoskins stated that each survivability test is looked at individually.

A. Bernhardt reviewed graphs correlating impacts to concentrations to determine if something is toxic, and said the graphs don't show a lot of correlation because there is not a lot of impact. A. Bernhardt stated that good correlations are needed to get cleanup numbers. There are not many samples with chemical concentrations exceeding their respective PECs. B. Hoskins said another approach would be to identify toxic samples (areas) instead of clean-up levels.

A. Bernhardt asked if we could use a higher threshold since this is not a high quality wetland. C. Rich said the data shows just a couple hot spots. K. Keckler said almost every sample is impacted to some degree (growth or survival). It was discussed whether or not improvement of the wetland can be justified under CERCLA.

Action Item:

- EPA to further review Area A Wetland data.
- A. Bernhardt to set up a conference call for April 1, 2009 to discuss this further.
- D. Conant (Navy) to explore non-CERCLA Navy funding options for improving the wetland.

Item 2: Outstanding Issues with Proposed Area A Wetland RI Structure

The second bullet was already discusses earlier in the day with C. Vu.

The only response that was discussed was for Comment #7. It was agreed that the Navy would conduct the risk assessment using a residential scenario.

Action Item:

- A. Bernhardt to modify the response to Comment #7.

Item 3: Discuss Structure/Status of Area A Wetland FS

K. Keckler indicated that she would ask D. Peterson whether the PECs needed to be added as TBCs.

Need to send the 100-year and 500-year floodplain map to CTDEP and EPA to determine whether floodplain ARARs need to be considered in the FS.

K. Keckler and M. Lewis agreed that if risk is removed, the wetland could be clean-closed.

K. Keckler suggesting looking at the EPA sediment guidance for other alternatives and suggested a combination of alternatives may be appropriate. She also suggested changing the natural attenuation with LUCs and monitoring to monitored natural recovery with LUCs.

K. Keckler indicated that she would discuss the levels of wetland replacement/restoration necessary for a removal action affecting the wetland. B. Hoskins and K. Munney said that there is no set number and depends on a variety of factors.

Action Items:

- K. Keckler to discuss ARARs with D. Peterson.
- A. Bernhardt to send out floodplain maps to CTDEP and EPA.
- K. Keckler to send information on wetland replacement guidance.

The meeting adjourned at 5 PM.

Summary of Conclusions:

- For the Draft Final Lower Subase Feasibility Study, Tetra Tech will revise the risk assessment using Oak Ridge 2008 values.
- For the Draft Final Lower Subase Feasibility Study, for TCE, Tetra Tech will revise the risk assessment using California EPA toxicity values for cancer slope factor and inhalation unit risk number, but use the NYS DOH non-carcinogenic value (10 $\mu g/m^3$).
- Collection of six additional samples from three locations at Inner Pier 1 sediment and analysis for PCB Aroclors is acceptable to EPA.
- The existing sampling and analysis plan for Thames River sediment can be amended to include the six additional Inner Pier 1 samples.
- The Inner Pier 1 Non-Time Critical Removal Action Work Plan will be revised to more clearly address turbidity monitoring and operational procedures, such as when the turbidity curtain can be opened, preventing "prop wash", coordination of the tug, barge, and turbidity curtain, and post-excavation confirmation sampling for the Outer Pier 1 area.
- Outer Pier 1 sediment exceeding criteria not removed during the Inner Pier 1 dredging will be handled under the Lower Subase FS.
- Outer Pier 1 sediment that exceeds either total ERM-Q>1.17 or PCB> 1 ppm must be addressed, including sediment at depths below 6 ft bss.
- The CERCLA dredging limit for Zone 4 will be included on figures provided in the Lower Subase FS.
- For soils in the Lower Subase Feasibility Study that are within the area defined by the risk/cleanup goal, if levels exceed TCLP or the 20 times rule-of-thumb regarding toxicity, then RCRA standards are relevant. High lead concentrations in saturated soil are not

under Connecticut jurisdiction, but as a hazardous waste issue it would still need institutional controls or a soil management plan to prevent digging.

Summary of Action Items:

- The Navy will look into possible additional funding to apply to dredging Outer Pier 1 sediment during Inner Pier 1 dredging.
- D. Conant will send M. Lewis and K. Keckler blind copies of the cover letter of the Corps of Engineers application for the maintenance dredging.
- C. Rich to send out tables with the revised ERM-Qs and an explanation of how the total ERM-Qs were calculated for both Pier 1 and Zone 4 areas.
- C. Rich to provide description of how the ERM-Q contour lines were generated on the sediment maps for both Pier 1 and Zone 4 areas.
- Navy maintenance dredging line to be shown on the Zone 4 sediment drawings.
- M. Lewis to provide written approval of the infiltration calculations by email.
- M. Lewis and R. Gatter-Evarts to review Attachment 1 of the Topic 2 - Item 1 handouts on copper and send their resolution to the Team by email.
- C. Rich to set up a conference call with M. Lewis and R. Gatter-Evarts for 9 AM on Wednesday, March 25, 2009 to discuss arsenic and copper issues.
- Navy to submit a request to CTDEP to formally approve the Alternative SWPC for phenanthrene of 23 µg/L.
- EPA to get back to the Team on the opportunity of reducing the number of ARARs tables.
- Tetra Tech to carry through the geostatistic method by the end of April to identify suggested sampling locations for the Zone 7 PDI.
- A meeting with the EPA to be held the end of April to review the results of the geostatistical Zone 7 PDI approach and compare those results to the EPA-suggested sampling and analyses.
- A. Bernhardt to modify the response to Comment #7 on the RI Outline.
- K. Keckler to discuss ARARs with D. Peterson.
- A. Bernhardt to send out floodplain maps to CTDEP and EPA.
- K. Keckler to send information on wetland replacement guidance.
- A. Bernhardt to set up a conference call for April 1, 2009 to discuss the Area A Wetland toxicity test results further.
- EPA to further review Area A Wetland data. Follow-up phone call scheduled for 1 PM on April 1 between Navy, EPA, CTDEP and Tetra Tech.

- D. Conant to explore non-CERCLA Navy funding options for improving the Area A Wetland.
- Possible Partnering Meeting on April 28, 2009 regarding the Zone 7 PDI and Inner/Outer Pier 1.

MEETING MINUTES FOR MAY 14, 2009 PARTNERING MEETING

Meeting Minutes
May 14, 2009 Meeting in Boston, MA
NSB-NLON, Groton, CT

Meeting Location: EPA Region I, Boston, MA

Agenda Topics:

1. Non-time Critical Removal Action Work Plan for sediment at Pier 1 Inner and Outer areas
2. Area A Wetland RI
3. Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)
4. Miscellaneous Issues and Meeting wrap up

Minutes Recorded by: N. Balsamo

Participants:

U.S. Navy (Navy): Dick Conant, Val Jurka, and Jim Gravette (new RPM)

U.S. Environmental Protection Agency (EPA) Region I: Kymberlee Keckler and Bart Hoskins

Connecticut Department of Environmental Protection (CTDEP): Mark Lewis

U. S. Fish and Wildlife Service (USFWS): Ken Munney

Tetra Tech NUS (TtNUS): Corey Rich, Nina Balsamo, Aaron Bernhardt, Jean-Luc Glorieux (by phone), and Betty Li (by phone)

Tetra Tech EC, Inc. (TtECI): Dan Sullivan and Roxanne Clarke

Gannett Fleming (GF): Greg Kemp and Todd Finlayson

Time of Meeting: 9:00 AM to 4:30 PM

Topic 1: Non-time Critical Removal Action Work Plan for sediment at Pier 1 Inner and Outer areas

- **Item 1. Review final draft of work plan**
- **Item 2. EE/CA Revision - Outstanding Issues**
- **Item 3. Discuss Plan of Action and Milestones for project implementation**

Copies of the Draft Final Non-Time Critical Removal Action Work Plan (NTCRA WP) for Sediment Removal at Pier 1 Inner and Outer Areas was passed out. V. Jurka reviewed the March 16 meeting discussion regarding additional characterization for waste disposal purposes: samples will be taken at three locations, at the top and bottom at each location, for a total of six samples. If one location cannot be sampled, it will be replaced with another location so that there will still be six samples, and all of the samples will be analyzed for PCB aroclors. V. Jurka stated that the original EE/CA included an estimate that the excavated sediment will be 20% hazardous and

80% non-hazardous, but that the Navy will do TCLP tests to improve the estimate for the amount of sediment that has the toxicity characteristic of hazardous waste. If the estimated amount of hazardous waste is less than EE/CA estimate, it is likely that additional money will be available to conduct dredging in Outer Pier 1.

Dick Conant stated that the Navy asked Nina Johnson to look for money to see if they can quickly modify the contract if necessary.

V. Jurka mentioned the sample acquisition strategy and that TtNUS was awarded the contract for the additional sampling.

D. Conant stated the Navy will try to dredge within the environmental window. First they need to finalize the work plan and EE/CA. Corey Rich stated he will do a brief work plan addendum, which is due to the Navy in June 2009. Hopefully sampling will be in July or August. Sediment samples will be tested for PCB aroclors and TCLP metals.

Action Item: TtNUS will prepare a brief work plan addendum for Inner Pier 1 waste characterization sampling.

D. Conant said that after the additional sediment sampling results are available, the Navy can determine how far dredging can go with the available funding. He also discussed the progress of the Pier 1 demolition project. The pile pulling is going well; the piles are not breaking off at the mudline. Pier 1 pile removal should be completed in 4 to 6 weeks.

D. Sullivan said he expects to get a sediment transportation and disposal proposal tomorrow. This proposal will help TtEC understand costs associated with the NTCRA.

G. Kemp requested that coordinates (northings and eastings) of existing and proposed sampling locations be provided to clarify descrepancies previously noted. V. Jurka suggested including them in the work plan. G. Kemp said that the dimensions of the Inner Pier 1 need to be clarified, as they are different in different documents. D. Conant said he would measure the Inner Pier, if necessary.

D. Sullivan asked K. Keckler if she had any comment on the Draft Final NTCRA WP to address in the Final version. K. Keckler mentioned that she would be happy with a tracked-change document. It would expedite EPA's review of the changes.

G. Kemp said we need to clarify northings, eastings and dimensions for a better estimate of volume. He said the 3rd paragraph on page 6, Section 3 was confusing as to when it refers to Inner Pier 1 and when it refers to Outer Pier 1. The main issue for the final NTCRA WP is turbidity monitoring. G. Kemp thinks background monitoring should be where the dredging will be conducted, not out in the river. Turbidity monitoring was further discussed. G. Kemp wants background turbidity readings taken in the work zone, then after dredging in the work zone. The turbidity meter would be placed inside where the curtain will go and monitored for 1 week (call it baseline, not background), then monitor to determine when to the open curtain. Water may be too shallow in Inner Pier 1 for an anchored turbidity meter while the project is going. When done dredging, use a hand-held meter to determine when to the open curtain. The group agreed to not have a buoy meter in the work zone during operations.

K. Keckler retrieved drawings showing scaled aerial photographs of the Lower Subase. C. Rich reviewed them versus drawings in the NTCRA WP and suggested the difference in the Inner Pier 1 length in different reports is the result of the correct footprint of the CIF building, which extends into the Inner Pier 1 area, not being correctly shown on the drawings. C. Rich suggested that the

NTCRA drawings should be updated using the aerial maps and said TtNUS would give the aerial maps to TtECI.

Action Item: TtNUS will provide available aerial mapping in electronic format to D. Sullivan at TtECI for use in updating WP figures.

The Team discussed characterization of excavated sediment and agreed that each barge needs to be fully characterized before it leaves the Subase.

D. Sullivan said the turbidity curtain will be anchored a minimum of every 50 ft. The curtain will be 2 to 3 ft longer than the depth at high tide. D. Sullivan said the fatal flaws with a clamshell are that the water is too shallow and the clamshell is not good in corners. The excavator will use a small bucket and during its use the operator will try to retrieve buckets filled with sediment and minimize the amount of water. The deck barges will have non-woven geotextile wrapped jersey barriers to dewater the sediment in the barges outside of the curtain. Water generated during the dewatering process will be allowed to drain directly into the Thames River. The barges will be moored until sediment is characterized. D. Sullivan does not yet know what disposal sites will be used for the sediment. A frac tank will be available as a "safety net", just for "nuisance water". If water is collected in the tank, it will be taken offsite for treatment and disposal. D. Sullivan said there is no room for the tug to go into the Inner Pier 1 area, but the deck-mounted excavator can move the barge itself.

C. Rich said TtNUS will use information from the NTCRA WP to revise and update the Inner Pier 1 EE/CA. The EE/CA will also evaluate the possibility of addressing portions of Outer Pier 1 during the NTCRA. G. Kemp said the NTCRA WP focuses on Inner Pier 1, but if sediment is also removed from Outer Pier , the WP needs to also address that. D. Sullivan said there would also be a turbidity curtain placed around Outer Pier 1 if dredging is conducted in that area.

G. Kemp said he had provided comments on the Final EE/CA after it went final. The Navy and TtNUS requested that the comments be forwarded to them so that they could be considered during revisisions to the EE/CA .

K. Keckler mentioned that their office is moving and she is in the process of sending boxes to the record center.

Navy and TtECI need draft EPA comments on the Draft Final NTCRA WP.

Action Item: EPA will provide draft comments on Draft Final NTCRA WP and comments on Final EE/CA.

TtECI/Navy will prepare a revised Draft Final NTCRA WP to address today's EPA concerns.

D. Conant asked M. Lewis about the Coastal Zone Form, CERCLA Exemptions. M. Lewis said the Navy should submit an application for informational purposes only. M. Lewis will provide a 401 Water Quality Certification template.

Action Item: M. Lewis to provide Navy with a 401 Water Quality Certification template.

M. Lewis stated that he had discussed sediment cleanup goals with Tracy lott in response to a request from C. Rich. The CTDEP would typically want sediments to be remediated to non-detect PCBs and an ERM-Q less than 0.5. The Team discussed that the goals proposed by the CTDEP were well below those established during the Thames River Validation Study and that Tracy lott was unfamiliar with the work that was done to establish the cleanup goals. The Team suggested setting up an informational call between the Navy and CTDEP to explain what was

done to date to determine the Thames River sediment cleanup criteria. B. Hoskins pointed out that if results are above certain screening levels, a baseline ecological risk assessment needs to be done, and it was done for this project and was used to develop the cleanup goals.

Topic 2: Area A Wetland RI

1. Discuss toxicity test results, preliminary remediation goals, and initial contamination nature and extent maps.

A. Bernhardt reviewed his handout, which was emailed to the Team prior to the meeting. The handout contained nine figures comparing PAHs, PCBs, DDT, and DDE results to EPA and Navy NOEC and LOEC values and geometric means of the NOEC and LOEC values; a table comparing EPA and Navy NOECs and LOECs; tables of sediment results, Threshold Effect Concentration (TEC) quotients, and Probable Effects Concentration (PEC) quotients; and a cover memo that discussed the figures and tables. A NOEC is the highest contaminant concentration that did not have an effect and a LOEC is the lowest contaminant concentration that did have an effect.

Table 2 bottom depths need to be corrected for some samples because sample bottom depths that say "4" are in inches, whereas other sample depths are in feet. A. Bernhardt, B. Hoskins, and K. Munney discussed the information and tried to resolve differences between the NOECs and LOECs suggested by the EPA and Navy. The primary issue relates to a disagreement over whether SD75 should be considered a toxic sample. B. Hoskins said one reference did well but one did not, so the fair way is to use a combined reference. The Navy does not agree because both reference stations were collected in areas with little contamination and even the reference station with lower survival and growth was not grossly impacted. Therefore, the Navy does not believe that a site sample should be expected to have better growth or survival than a reference station.

A. Bernhardt said if you compare figures, the areas are not that much different whether using the EPA or Navy NOECs and LOECs. J. Gravette said the PAH values looked low compared to Region 3 standards.

K. Keckler would be interested in a map with all the EPA and Navy NOECs and LOECs overlaid to easily see how they compare. A. Bernhard planned to determine the methodology first. B. Hoskins noted that sample location 2WSD66 was toxic to *Hyalella azteca* and 10 TEC-Qs were exceeded at this location, so he thinks that sediment should be removed from that location. B. Hoskins was considering a criterion of 8 to 10 TECs exceedances as showing a potential ecological concern. A. Bernhardt noted that reference locations 82, 83, and 84 have 7 to 8 TECs exceedences (but no toxicity test data) so 8 TEC exceedences is too conservative. B. Hoskins asked how big the circles around the exceedance locations are, and suggested bounding using the mid-point between exceedance and non-exceedance locations or kriging.

B. Hoskins said it is easier to have fewer cleanup goals. G. Kemp said it is hard to limit cleanup goals if there are 10 TECs. K. Keckler inquired about toxic status. A. Bernhardt said he is using chemical concentrations as criteria, not toxicity, because you need typically need chemical concentrations for cleanup numbers, although you could have a cleanup goal based on the number of TEC exceedences.

B. Hoskins said the issue is not just excavation of sediment but wetland restoration – it doesn't make sense to leave phragmities between restored areas. It would be good to know where to stop excavation before going out to complete the remediation. D. Conant stated that whether or not to backfill excavated sediment has not yet been determined.

The areas outlined in orange on Figures 1 and 2 were compared and it was determined that the yellow area (greater than NOEC but less than LOEC) should not be included within the orange outlined area.

The team could not reach consensus on the major issues during the meeting; therefore, a follow up conference call was scheduled.

Action Item: A follow-up call on Area A Wetland is scheduled for May 27, 2009 at 10:00 AM.

Action Item: A. Bernhardt to add the meaning of the line colors to the legend of the wetland figures and not include yellow area within red area on Figure 1.

Topic 3: Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)

Item C: Review of final revised tag maps and extent of contamination diagrams for Zone IV and Pier 1. (To address to Items A & B later.)

C. Rich passed out a handout with a cover memo, ERM-Q calculations, and 14 tag maps. This handout had been emailed to the Team prior to the meeting. K. Keckler had previously sent an email saying she was fine with the calculations. Since the calculations had not changed since April, C. Rich discussed the tag maps, noting that the purple area at Outer Pier 1 and the pink area on the Zone 4 figures were artifacts from the Validation Study and were no longer considered as the remediation areas. D. Conant said that the Pier 6 dredging boundary shown on the Zone 4 figures may not be accurate. For total ERM-Qs at Outer Pier 1, C. Rich noted that the area expands with depth, and that at each depth the ERM-Q area more than covered the PCB areas. C. Rich said dredging the top 2 feet and placing clean fill would alleviate ecological risks, whereas removing all contaminated sediment would eliminate ecological risks and land use controls (LUCs). The team needs to resolve remediation goals and areas now to allow the EE/CA and FS to proceed. Future use issues will be determined during the feasibility study.

C. Rich then reviewed the Zone 4 tag maps, noting the since the FS, the estimated sediment remediation volume in Zone 4 increased to 11,000 cubic yards. Similar to Outer Pier 1, the ERM-Q exceedance area increases with depth and the ERM-Q area generally covers the PCB area at Zone 4.

K. Keckler asked how deep the bedrock is at the dredge buffer. D. Conant estimates the bedrock is 50 to 60 feet at that location.

K. Munney said he did not review the Validation Study (where the ERM-Q of 1.17 was determined), but he reviewed the ERM-Q calculations and in some Zone 4 locations PAHs were 2 to 5 times greater than ERMs. He asked the team if this was accounted for in toxicity testing. B. Hoskins thinks the high PAHs get averaged away. C. Rich said there were no clear results in the toxicity tests and that locations of concern with high PAHs generally get captured inside the dredge buffer line. V. Jurka said that they did reduce the number of COCs to the primary COCs during the Validation Study and that some PAHs may not be bioavailable.

C. Rich asked if the ERM-Q of 1.17 is the correct number. K. Keckler said yes. C. Rich asked if the Navy can use these ERM-Qs/calculations/ figures to move forward with the FS. The Team decided to discuss this topic again on May 27, 2009 in light of information provided by CTDEP and questions asked by K. Munney.

B. Hoskins said that ERM-Qs were acceptable for Pier 1 and the Navy can go ahead with the Pier 1 EE/CA. For Inner Pier 1, the NTCRA will remove sediment to bedrock.

Action Item: K. Munney to review Zone 4 sediment ERM-Q information. The acceptability of the selected ERM-Qs and tag map areas for Zone 4 will be discussed during the May 27, 2009 conference call.

D. Conant to provide the updated dredge boundary for Pier 6 to TtNUS.

Topic 3 Item B: Pre-Design Investigation "strawman" for Zone 7 lead

N. Balsamo passed out the handouts, which included a summary of the four-step method of planning the investigation, six pages to explain the geostatistical method used, and 28 figures. These handouts had been emailed to the Team prior to the meeting. N. Balsamo reviewed the first steps of planning the investigation, which included reviewing site history and comparing boring logs to analytical results. B. Li reviewed the geostatistical method, and N. Balsamo reviewed relevant regulatory requirements and how the information was combined to select the boring and sample locations. Under the industrial/commercial scenario, soil that exceeded 1,090 mg/kg in the top 2 feet beneath pavement would be remediated. For the residential scenario, mass lead greater than 400 mg/kg was relevant to a depth of 15 feet to determine the area of risk.

G. Kemp said that the geostatistical approach to developing the sampling plan was acceptable, but since the Navy will actually remediate to the industrial/commercial scenario, not the residential scenario, that it would be more cost effective to do fewer deep samples and more surface samples. C. Rich said the area greater than 400 mg/kg needs to be established to determine the area with LUCs; G. Kemp said LUCs are already needed wherever we only remediate to 1,090 mg/kg in the top 2 feet.

M. Lewis said this is one of the more rigorous approaches he has seen but he does expect some degree of understanding of concentrations to 15 feet. K. Keckler asked if there are concentrations the CTDEP would not allow leaving in place to depth. M. Lewis said probably not.

J. Gravette suggested removing 7PDI 10 and 7PDI11 because the age of the buildings preceded ash incineration. D. Conant asked if the figures could be changed to show reduced uncertainly beneath Buildings 157, 106 and 103 for the same reason.

C. Rich said this PDI method will be used to develop sampling plans for all the zones. The three dimensional kriging maps will help to refine the volume estimates for the FS. The FS will proceed with the data that what we have, and the draft PDI work plan will be done before the FS is completed. The data collected under the PDI Work Plan will be incorporated into future documents.

C. Rich mentioned that the uncertainty model could be revised based on field XRF testing and the uncertainty analysis and kriging would be revised based on the PDI results.

Action Item: The Team is to conduct a meeting to determine which contaminants will be further investigated in each zone.

Topic 3 Item A. Copper and Arsenic in LB groundwater. Status of groundwater resampling effort.

C. Rich passed out the handout which he had previously emailed to the Team before the meeting. The Navy will conduct two rounds of sampling to address CTDEP's concerns with

copper and arsenic in Lower Subase groundwater. The Navy has funded TtNUS to conduct the sampling. TtNUS plans to complete the work plan ASAP for early fall sampling, to be followed by the second round next spring.

The handout included a summary table of the proposed groundwater monitoring wells to be sampled on a zone-by-zone basis, based in part on which wells had previous detections greater than the SWPC. The table includes two Zone 4 wells (WE5 and 19MW4) that were destroyed. If necessary, those locations could be sampled using temporary wells. G. Kemp said the arsenic in Zone 1 could be related to Building 29.

M. Lewis said he needed a day or two to think about the proposed sampling. The selected wells will be discussed during a conference call scheduled for May 20, 2009. He will email a link of CT guidance for groundwater monitoring, but said the state has no specifics on temporary wells.

Action Item: Navy and TtNUS will conduct a conference call with M. Lewis/CTDEP to discuss selected wells for arsenic and copper monitoring on May 20, 2009. M. Lewis will email a link for CT guidance on groundwater monitoring to team.

Topic 4: Miscellaneous Issues and Meeting wrap up

1. Groundwater monitoring - DRMO/Area A/Goss Cove
2. Status of NAVFAC Groundwater Monitoring and Landfill Cap O&M contract
3. RAB Newsletter and tentative meeting
4. SUBASENLON IR instruction status

K. Keckler mentioned that we are close to deleting the base from the NPL, and would like to celebrate this with the community.

Navy said the last item the Navy owes on OU9 is the plat map and Class A2 survey for the ELUR. M. Lewis said it should have the meets and bounds survey of the restricted area in terms that are obvious in real life for a lay person, such as buildings.

TtNUS to provide a revised IR Instruction figure to the Navy.

D. Conant suggested a production call in mid-June rather than meeting again in Boston. K.Keckler would like a clear agenda before then.

Action Item: Technical /RAB (evening)/ Wetland meetings scheduled for July 15, 2009. The technical meeting will be conducted at 1300 at NSB-NLON.

C. Rich said the human health risk assessment (HHRA) for the feasibility study will be ready in the next week or so. Should we submit this to Chau Vu now (through K. Keckler)? K.Keckler said yes.

Action Item: TtNUS to submit HHRA to EPA for review prior to proceeding with the FS.

The next version of the Lower Subase Feasibility Study will be draft final.

C. Rich asked how to prepare a final set of response to comments for the FS. Should we include all meeting minutes? K..Keckler said yes. She has no preference on how it is done. J. Gravette suggested chronologically.

G. Kemp returned to the topic of the PDI "strawman". TtNUS's proposed PDI has only SPLP for additional leachate testing – do we need TCLP testing? K. Keckler said it depends on why you are sampling. G. Kemp said the zones will be paved anyway, which in his mind constitutes a cap and there will be LUCs anyway. K.Keckler said she will talk to Dave Peterson.

Action Item: K. Keckler to talk to Dave Peterson about the need for additional TCLP testing for the soil PDI.

D. Conant will take photos of the Pier 1 demolition and get them to the Team. He doesn't need to measure Inner Pier 1 because the aerials provided the needed information.

The meeting adjourned at 4:30 PM.

Summary of Conclusions:

- ERM-Qs were acceptable for Pier 1 and the Navy can go ahead with the Pier 1 EE/CA.

Summary of Action Items:

- TtNUS to do brief work plan addendum for Inner Pier 1 waste characterization sampling.
- EPA to provide draft comments on Draft Final NTCRA Work Plan.
- EPA to provide the post-final comments on the Final EE/CA.
- M. Lewis to provide the Navy with a 401 Water Quality Certification template.
- The next version of the NTCRA Work Plan will again be Draft Final. Corrections will be provided in track-change mode.
- TtNUS will provide aerial mapping of Pier 1 area to D. Sullivan.
- An informational call needs to be set up between the Navy, B. Hoskins and CTDEP (M. Lewis and Tracy Iott) to discuss how the sediment ERM-Q and PCB criteria were determined.
- For the Area A Wetland handout – TtNUS to correct the bottom depths on Table 2 so that they are all in the same units, label the figure line colors in the legend, and remove the yellow area from the orange-outlined area in Figure 1.
- A follow-up call on Area A Wetland is scheduled for May 27, 2009 at 10 AM.
- A conference call will be conducted on May 27, 2009 to discuss whether the Navy can move forward using the ERM-Q criterion of 1.17 and the calculations and figures provided for Zone 4.
- A meeting needs to be scheduled to plan which contaminants will be further investigated in the PDI for each zone.
- A conference call is scheduled for May 20, 2009 at 3:00 PM with M. Lewis/CTDEP to discuss selected wells for arsenic and copper monitoring.

- D. Conant to provide the updated dredge boundary for Pier 6 to TtNUS.
- M. Lewis to email a link for CT guidance on groundwater monitoring.
- A Team production call is tentatively scheduled for June 17, 2009. Need to prepare and provide an agenda.
- TtNUS to submit the HHRA to EPA for review prior to proceeding with the FS.
- Technical /RAB (evening)/ Wetland meetings are scheduled for July 15, 2009. The technical meeting will be at 1:00 PM at NSB-NLON.
- K. Keckler to discuss with Dave Peterson whether there is a need for TCLP testing as part of the soil PDI.

Rich, Corey

From: Keckler.Kymberlee@epamail.epa.gov
Sent: Wednesday, June 17, 2009 3:35 PM
To: james.gravette@navy.mil; richard.conant@navy.mil; val.jurka@navy.mil; Rich, Corey; gkemp@gfnet.com; rtfinlayson@gfnet.com; mark.lewis@po.state.ct.us; Kenneth_Munney@fws.gov
Subject: Draft Minutes for the May 14, 2009 Boston meeting - Lower Subase FS/Pier 1 RAWP/ Area A Wetlands

EPA reviewed the draft minutes for the May 14, 2009 meeting in Boston regarding the Lower Subase Feasibility Study (FS), the Pier 1 Remedial Action Work Plan (RAWP), and the Area A Wetlands investigation for the Naval Submarine Base in Groton, Connecticut.

TOPIC #1: Non-time Critical Removal Action Work Plan for sediment at Pier 1 Inner and Outer areas

1. p. 2 of 9: Turbidity Monitoring: EPA does not concur that the group agreed not to have a buoy meter in the work zone during operations because no agreement on the use of a hand held meter was reached during the meeting. TtEC agreed to provide additional information regarding the hand held meter proposed for use and the procedure for taking measurements. The Navy agreed to provide this information in a revision of or supplement to the RAWP (or in advance). Also the stated intent in the RAWP to have the remediation contractor take turbidity readings is not acceptable. Collection of turbidity readings and the excavation work should be performed by independent entities. It may be more appropriate to collect turbidity readings automatically in the work zone to avoid potential conflict of interest issues. EPA will review Navy's turbidity monitoring information when received and additional discussion can be held to resolve this issue. (This was resolved on June 17, 2009 – OK to sweep using a hand-held but it must be done at at least two different depths)

Aerial Extent of Remediation and Drawing Revisions: Because the current RAWP does not account for the removal of Pier 1, the aerial extent of remediation for the Inner Area and the associated drawings in the RAWP do not accurately reflect the conditions that will be encountered when the remediation is conducted. Discussions during the meeting resolved that the drawings in the RAWP need to be updated to reflect the removal of Pier 1 before initiation of the remedial action. This is significant because it impacts deployment of the turbidity curtain, the aerial extent of removal (sediment beneath the pier needs to be addressed), and potentially the number and location of the turbidity monitoring stations required. The Navy agreed to address this issue by revising the RAWP to reflect the removal of Pier 1 and to update the drawings and text as appropriate.

TtEC agreed to provide a red-line strikeout revision of the RAWP to expedite EPA's review of the proposed revisions. (EPA recognizes that this has been captured in the meeting summary section.)

2. p. 3 of 9: EPA Comments on the RAWP: The minutes should reflect that EPA distributed draft comments on the draft final RAWP during the meeting. TtEC was to use those comments together with the meeting discussion/resolutions to prepare a red-line strikeout revision of the RAWP.

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MEETING MINUTES FOR JUNE 17, 2009 CONFERENCE CALL

Meeting Minutes
June 17, 2009 Conference Call
NSB-NLON, Groton, CT

Agenda Topics:

1. Non-time Critical Removal Action for sediment at Pier 1 Inner and Outer areas
2. Area A Wetland RI (resolved prior to call so not addressed)
3. Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)
4. Meeting wrap up

Minutes Recorded by: N. Balsamo

Participants:

U.S. Navy (Navy): Dick Conant, Jim Gravette, Val Jurka, and Jennifer Wright

U.S. Environmental Protection Agency (EPA) Region I: Kymberlee Keckler

Connecticut Department of Environmental Protection (CTDEP): Mark Lewis

Tetra Tech NUS (TtNUS): Corey Rich, Nina Balsamo, and Jean-Luc Glorieux

Tetra Tech EC, Inc. (TtEC): Dan Sullivan and Roxanne Clarke (Topic 1)

Gannett Fleming (GF): Greg Kemp and Todd Finlayson

National Oceanic and Atmospheric Administration (NOAA): Ken Finkelstein (Topic 1)

Time of Meeting: 10:00 AM to 12:30 PM

Topic 1: Non-time Critical Removal Action (NTCRA) for sediment at Pier 1 Inner and Outer areas

A. Progress on final draft of work plan (WP)

D. Conant stated that internal efforts to secure additional funding were received for this project so now the budget covers the Outer Pier 1 (OP1) dredging to a depth of 6 feet. Whether or not OP1 is included in the Feasibility Study (FS) depends on what sediment is left behind after dredging. There aren't sufficient funds to address the "bubble area" [a location 4 to 6 feet deep covered by cleaner sediments, south of former Pier 1 (approximately 14,400 square feet)]. The "fan area" emanating from Inner Pier 1will be covered by the EE/CA. As a result of the additional funding, the dredging Work Plan will need to be revised to reflect the additional efforts.

D. Sullivan said TtEC is working on the UFP-SAP and QAPP, which should not hold things up. They are getting pricing for the changed dredging plans. The work plan will now include OP1, rather than having OP1 as an addendum. Because adding OP1 will result in many changes to the work plan, they will not track changes, but will highlight changes to address meeting comments.

G. Kemp wants more information on the turbidity method protocol. He prefers turbidity automatically recorded, not manually monitored by the dredging contractor (potential conflict of

interest). G. Kemp also had issues with some of the minutes from the May 14 meeting (his comments have not yet been passed along to the Team).

D. Sullivan said he cannot have a turbidity monitoring buoy in the work zone without destroying it because of the limited space. R. Clarke said she will include more information from the vendor to compare hand held versus buoy monitoring. R. Clarke indicated that the identical technical sonde internals will be used in the hand held as the buoy so that same readings would be obtained regardless of being hand held or buoy. The Team discussed hand-held versus buoys, one versus two meters and multiple depth measurements. G. Kemp wants water levels at critical points and would be okay with two hand held meters at different depths, but thinks that one depth is not enough in open water. R. Clarke said they could do a sweep with one monitor, just use more cord to take multiple depths with a single hand-held meter. G. Kemp said okay as long as there are good readings before they open the curtain.

R. Clarke said the SAP will be an attachment to the WP. The goal is to get the SAP and WP to the Navy by July 10, then to EPA and CTDEP by the end of July.

J. Gravette suggested 5-point (four corners and the center) composite confirmatory samples on the excavation bottom, and sample for PCBs where appropriate and ERM-Qs. D. Sullivan suggested one composite sample every 5,000 square feet based on previous work (McAllister Point). K. Keckler said every 5,000 square feet sounds too high. After further discussion, the Team, including K. Keckler, agreed to sample 9 points per 5,000 square feet (about a 70 ft x 70 ft area).

Based on previous data, the excavation sides will not need confirmation sampling. For perimeter sampling J. Gravette said we have data that shows 2 of 3 sides are clean. G. Kemp said if the sidewall is half way between clean and a hit, the Navy needs to show that it is clean.
D. Sullivan estimated a total dredging volume of 7,700 cubic yards (6,000 cy to 6 feet plus 1 foot over-dredge plus 700 cy to slope side walls to 1.5:1.) The OP1 dredging area (without the separate contaminated area to the south) was estimated at 0.6 acres.

D. Sullivan said he will leave it up to the dredger whether or not to use an excavator or clam shell bucket in the OP1 area. D. Conant said if they switch to a bucket and 1 foot of over-dredge, that needs to be included in the work plan.

Possible NTCRA WP schedule – Draft to regulators at end of July, regulatory comments due end of August, response to comments by mid-September, and final report out end of September. K. Keckler said that schedule is missing the draft final stage.

D. Conant said if our goal is October 1, that allows 2 months of work in a 4 month environmental window.

D. Sullivan said the original scope was 2 months, but since 5,000 cy was added, it now looks like 10 weeks of dredging. October 15 is as tight as they can go without a variance from the environmental dredging window. D. Conant thinks we can get a variance based on earlier discussions with the State based on the use of turbidity curtains

Topic 1: Non-time Critical Removal Action for sediment at Pier 1
Inner and Outer areas

B. EE/CA Revision - Outstanding Issues (ERM-Q finalization and scope/costs of project)

TtNUS had previously started to do the EE/CA last December/January, but found that major issues on OP1 had to be resolved before we could proceed. C. Rich said TtNUS is just starting to

re-initiate the EE/CA effort and had some questions. Will this be an addendum EE/CA or a complete revision of Battelle's 2008 EE/CA? Due to OP1, he expects that the technology and ARARs will not change significantly, but that the volume and costing will require significant revisions. We need an Action Memo before October 1 for the NTCRA. What is the Team looking for in an addendum?

K. Keckler said there were a number of outstanding issues on Battelle's EE/CA. She will summarize the issues in a November 2007 letter and email them.

C. Rich to check 2008 EE/CA appendix response to comments to see if November 2007 comments and January 2008 responses were included.

C. Rich and J. Gravette stated that Action Memos are typically Navy documents signed by only the Commanding Officer, but the EE/CA is for public comment and needs a Public Notice. The following possible schedule was discussed: revised EE/CA to the Navy by the end of July, to regulators for review in August, to be finalized by the end of September. J. Gravette needs to put together a schedule for the WP and EE/CA, since they are inter-related. He will send the schedule to C. Rich and D. Sullivan.

Topic 2: Area A Wetland RI/FS

This topic was not discussed during the conference call. It was rescheduled and will be addressed during a conference call on June 25, 2009 at 11:00 AM.

Topic 3: Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)

A. Status of Copper and Arsenic in LB groundwater resampling effort

C. Rich stated that at the May 14, 2009 meeting, he provided a summary of the groundwater issues and a proposal for resampling. On May 20, the proposal was discussed on a conference call with CTDEP, and the State agreed with most items. The Navy proposed 15 wells and the State asked for one more, for a total of 16 wells to be resampled. Two of those wells will need to be reinstalled, probably with permanent wells so they can be sampled more than once. The plan is to do two rounds of sampling (this fall and next spring to get varied hydrologic conditions) in Zones 1, 4, 5, and 7 wells.

TtNUS is working on a UFP-SAP for this groundwater sampling. The goal is to get this SAP to the Navy in mid-July and the regulators at the beginning of August. In addition, the Inner Pier 1 sediment SAP was issued to the Navy today and the soil PDI work plan is due to the Navy at the end of July. D. Conant said that we will be looking for comments for the State on these reports. M. Lewis asked to receive 2 copies of the groundwater UFP-SAP since the reports will be reviewed by both he and Rosemary Gatter-Evarts. J. Gravette will alert the Navy chemist to expect these UFP-SAPs.

Action Items:

- TtNUS to send 2 copies of groundwater UFP-SAP to CTDEP.
- J. Gravette to alert Navy chemist to expect UFP-SAPs for Inner Pier 1 sediment (now) groundwater sampling (mid-July), and soil PDI (late July).

Topic 3: Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)

B. PDI proposal for all LB zones (handout emailed prior to call)

N. Balsamo (TtNUS) reviewed the general approach for developing the PDI. TtNUS is applying the approach presented at the May 14, 2009 Team meeting for Zone 7 soil lead to TPH and PAHs in Zone 7 and to lead, TPH and PAHs in Zones 1 through 4. Since they are connected, Zones 1 through 4 will be kriged together to maximize the use of data. COCs, regulatory criteria, and geostatistical methods were reviewed.

G. Kemp commented that the handout showed that industrial criteria are applicable to a depth of 2 feet under pavement, but that we would need to re-assess the industrial scenario if the Navy decides to remove pavement. That would also affect the infiltration rate for the Alternative PMC calculations. D. Conant said he does not foresee removing pavement – although it is always a possibility, it is a safe assumption that paved areas will remain paved. D. Conant also said if we change from pavement, the Navy needs to review assumptions. G. Kemp said the report needs a paragraph to that affect.

M. Lewis commented that for the direct exposure criteria, under the industrial scenario, if soil is remediated to 2 feet under pavement, the site will still require LUCs for remaining contaminated soil to a depth of 15 feet. For the residential direct exposure scenario, contaminated soil can be left in place if LUCs are applied to a depth of 15 feet. G. Kemp stated that remediation to the residential scenario is unlikely and in his opinion the most effective use of resources would be to focus on industrial risk. The Team discussed this and the Navy confirmed that regardless of regulatory requirements, Navy guidance requires evaluating the clean closure scenario.

N. Balsamo said that for Zones 1 through 4 and 7, geostatistics will be used to compare to direct exposure criteria (DEC) and PMCs to TPH and BAP data to determine additional sampling locations. Geostatistics will also be used to compare to direct exposure criteria DEC to lead data to determine additional sampling locations. There is not enough data to use geostatistics for Zones 5 & 6 or for SPLP results. G. Kemp asked what will be done instead of geostatistics for SPLP testing. C. Rich said the proposed sampling locations will also be sampled for SPLP. G. Kemp asked if after Zones 5 and 6 are resampled, if there would be enough data then to do geostatistics. N. Balsamo said that will be considered, either that or new sample results may bound the previous hits with clean results.

N. Balsamo stated that benzo(a)pyrene (BAP) equivalents will be used to represent all carcinogenic PAHs. G. Kemp said that BAP equivalents are for risk assessments, but we need to look at each contaminant and screen each PAH against each PMC. M. Lewis confirmed that we need to compare all PAHs to each corresponding PMC.

N. Balsamo said one proposed use of the results would be to recalculate geostatistics to refine the remediation areas and volumes for the Remedial Design. G. Kemp asked how the "grey" areas would be handled after the geostatistics are rerun. C. Rich said that is a step ahead of what we are doing, to be decided during development of the Remedial Action Work Plan.

G. Kemp asked if uncertainty for each COC would be addressed in the combined uncertainty maps. N. Balsamo said yes.

N. Balsamo explained that geostatistics does not consider some site-specific conditions. For example, the direction of groundwater flow might prevent TPH from spreading east, and if the railroad embankment was constructed prior to base activities, soil contamination could not occur

beneath the railroad embankment. D. Conant said he has a cultural resources survey that states that the railroad embankment was built in 1898. The Navy yard was started in 1868, and he has the base boundary for 1915. He will send this information to TtNUS to incorporate into the PDI planning.

G. Kemp asked how we were planning to handle TCLP testing. J. Gravette commented that there will be a meeting in mid-July for Navy and EPA legal personnel during which TCLP policy will be discussed.

Topic 3: Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)

C. Resolve issue of elevated PAHs in Thames River Zone IV sediments (Recap outcome of tentative call planned with EPA/USFWS/CTDEP 2nd week of June).

This issued was previously resolved. K. Keckler will forward K. Munney's email to G. Kemp. K. Keckler said K. Munney retracted his concerns about revisiting the ERM-Qs. C. Rich said now we can re-initiate the sediment portion of the FS. D. Conant said we still need to resolve the PCB concerns from the May 14 meeting. Tracy lott and Rosemary Gatter-Evarts (CTDEP) and Bart Hoskins (EPA) are to discuss the PCB issue. K. Keckler said we do not need to wait for them. D. Conant said we need to resolve State concerns.

G. Kemp asked what the Navy plans to do along the Quay Wall during river sediment remediation. C. Rich said the details will be handled in the Remedial Action Work Plan, not the Feasibility Study. J-L Glorieux said we will proceed with the FS on the assumption that we can remove sediment to a depth of 6 feet at the Quay Wall on a temporary basis. The 6 foot depth would remove all known exceedance soil, subject to verification sampling. D. Conant reiterated that he and TtNUS had discussed this approach with the engineers at the Public Works Department at NSB-NLON. The engineers had indicated that this was a viable approach.

The Team discussed how to handle OP1 sediment in the FS. The FS will ignore the NTCRA and assume no sediment has been removed from OP1. JL Glorieux noted the role of the FS is not to make recommendations, just to evaluate alternatives. C. Rich said we could capture all of the OP1 sediment in the FS and present alternatives. When the ROD is written, we can capture that the NTCRA was done and would need to redo the alternative for the ROD. The Team discussed whether the area at TRP1-SD-005 (a.k.a "the bubble area") should be handled separately in the FS. K. Keckler said that may save time in the long run. TtNUS and Navy to discuss.

Topic 3: Lower Base FS - Follow-up issues from 16 March meeting (Remaining outstanding issues)

D. FFA schedule update

J. Gravette mailed an extension request on June 4. K. Keckler, M. Lewis, and D. Conant did not receive it yet. J. Gravette will scan the document and send to K. Keckler, M. Lewis, D. Conant and C. Rich. The Letter will include an updated schedule for the Lower Subase FS and Area A Wetland.

Topic 4: Meeting Wrap-up

A. Review action items

D. Conant reviewed action items with Team (see IR tracker spreadsheet).

K. Keckler said that depending on the results of the July legal meeting, if there are no TCLP tests samples that fail the 20 times rule of thumb rule will be targeted as hazardous and be targeted for remediation. J. Gravette said this is hard to accept for samples that pass residential soil screening levels. To be discussed in July.

D. Conant said he did a manhole inspection and will send a memo on it. D. Conant sent Pier 1 demolition photos. K. Keckler said she received them. D. Conant said the new Base Instruction has been signed and he emailed it to Team members. K. Keckler would like a paper copy of the new Base Instruction.

J. Gravette to send out a PDF of the FFA schedule extension request.

J. Gravette to prepare a Plan of Action and Milestones (POA&M) for the NTCRA and EE/CA.

D. Conant to send a copy of the cultural resources report to TtNUS.

C. Rich mentioned that TtNUS is still waiting for input from CTDEP on specific monitoring criteria for the O&M Manual.

Topic 4: Meeting Wrap-up

A. Confirm RAB/Tech Meeting/Area A wetland tour for mid-July

Team agreed that the RAB/Tech Meeting/Area A Wetland tour will be conducted on July 16, 2009. The Tech Meeting or Area A Wetland tour will be conducted in the morning and afternoon, and the RAB meeting will be in the evening.

Summary of Action Items:

1. TtEC to provide more information on turbidity monitoring for the NTCRA.
2. TtEC to provide a SAP with NTCRA WP.
3. C. Rich to check 2008 EE/CA appendix Response to Comments to see if November 2007 comments and January 2008 responses were incorporated.
4. When the groundwater SAP is done, Navy to send two copies to CTDEP (for review by M. Lewis and R. Gatter-Evarts).
5. J. Gravette to alert Navy chemist that he will be receiving three UFP-SAPs from TtNUS between now and the end of July.
6. TtNUS to include a paragraph in the FS that under the industrial scenario, if the Navy removes pavement, the alternative assumptions should be reviewed.
7. The Lower Subase Remedial Action Work Plan (following the PDI) to screen each PAH against its corresponding individual criterion, rather than using the BAP equivalent.
8. D. Conant to send the cultural resources survey to TtNUS for consideration in development of the PDI SAP.

9. K. Keckler to forward K. Munney's email to G. Kemp regarding PAHs in Zone 4 sediments.

10. Tracy Iott and Rosemary Gatter-Evarts (CTDEP) and Bart Hoskins (EPA) are to discuss the sediment PCB issue.

11. TtNUS and Navy to decide how to handle OP1 sediment in the FS – evaluate as a single area or divide into the sediment adjacent to Inner Pier 1 and a separate area at TRP1-SD-005 (a.k.a. "the bubble area")?

12. J. Gravette to scan the schedule extension request, including updates for the Lower Subase FS and Area A Wetland, and send to K. Keckler, M. Lewis, D. Conant and C. Rich.

13. D. Conant to send a memo on the manhole inspection he conducted in the Lower Subase.

14. D. Conant to send K. Keckler a paper copy of the new Base Instruction.

15. J. Gravette to prepare a Plan of Action and Milestones (POA&M) for the NTCRA and EE/CA.

16. CTDEP to provide previously requested monitoring criteria for the O&M Manual to TtNUS.

APPENDIX F

RESPONSES TO EPA COMMENTS

Responses to EPA's July 23, 2009 Comments
Draft Inner Pier 1 Sediment UFP-SAP
NSB-NLON, Groton, Connecticut

Initial Issue: August 12, 2009
Revised: August 31, 2009

GENERAL COMMENTS

General Comment 1:

Since the removal action will include most of the Outer Pier 1 also, EPA recommends that the Sampling and Analysis Plan be expanded to include this area as well.

Response:

Disagree. The focus of this UFP-SAP is Inner Pier 1. Additional sampling is required in Inner Pier 1 to characterize the sediment for disposal purposes. Outer Pier 1 sediment was sampled in October 2008 and analyzed for PCB – Aroclors, TCLP Metals, and other parameters. The Outer Pier 1 analytical data indicated that PCB – Aroclors and TCLP metal concentrations were well below TSCA and hazardous waste identification levels, respectively. Therefore, no additional sampling is needed in Outer Pier 1.

August 20, 2009 Conference Call Resolution:

A UFP-SAP that covers confirmation sampling for Outer Pier 1 was prepared by Tetra Tech ECI and included in the Draft Final Non-Time Critical Removal Action Work Plan for Inner and Outer Pier 1 (August 6, 2009). Preparation and submittal of this UFP-SAP addressed EPA's concern. No further action is required.

General Comment 2:

Please clarify how subsurface samples will be collected if the sediment depth is less than four feet. Would Navy collect subsurface sediment samples from the two to three foot depth range? or abandon collecting subsurface sediment at that or a nearby location with the same sediment depth?

Response:

The plan as presented on p. 35 and p. 45 of the SAP will be to collect surface (0 to 1 foot) and subsurface (2 to 4 feet) samples from locations TRP1-SD-11 through TRP1-SD-13. If refusal is encountered prior to reaching approximately 3.5 feet below sediment surface, than no subsurface sample will be collected from that location, but one will be collected at one of the other four proposed sediment sample locations (TRP1-SD-14 through TRP1-SD-17) which are expected to have thicker sediment.

The reference to the 3.5 foot depth requirement will be added to the text on p. 35 and p. 45 of the SAP.

August 20, 2009 Conference Call Resolution:

EPA inquired what the Navy would do if refusal is encountered prior to reaching approximately 3.5 feet below sediment surface at all three locations (TRP1-SD-11 through TRP1-SD-13). The Navy responded that all three subsurface samples will be collected as appropriate from the other four proposed sample locations (TRP1-SD-14 through TRP1-SD-17). The text will be clarified as necessary.

SPECIFIC COMMENTS

Specific Comment 1: p. 11, Worksheet #3

Bonnie Capito is listed twice.

Response:

Agree. The second entry on p. 11 will be deleted.

Specific Comment 2: p. 26, § 10.1.3

The last sentence at the top of the page states that no further activities are planned for the sediment under Pier 1. Figure 4 in the Revised Draft Pier 1 Work Plan indicates that sediment beneath the former Pier 1 will be dredged during the Outer Pier 1 removal action. Please ensure consistency with the work plan.

Response:

The sentence will be changed to the following:

"All Inner Pier 1 sediment and a majority of the contaminated Outer Pier 1 sediment will be remediated through a Non-Time-Critical Removal Action (NTCRA). The contaminated sediment remaining in Outer Pier 1 after the NTCRA will be addressed under a different remedial strategy determined through the Lower Subase Feasibility Study and subsequent Proposed Plan and Record of Decision."

Specific Comment 3: p. 28, § 11.1

The last sentence should state that PCB-contaminated sediment must be disposed based on its in situ PCB concentrations. Please edit accordingly.

Response:

The following sentence will be added at the end of the paragraph:

"…waste landfill. If the sediment meets the requirements of a solid waste landfill, the contaminated sediment can be disposed in this type of landfill; otherwise, the sediment will need to be further characterized ex-situ and then treated and/or disposed in a hazardous/TSCA waste landfill. For the PCB-contaminated sediment, it will be disposed based on its in-situ concentrations."

Specific Comment 4: p. 35, Worksheet #14

a) To properly evaluate the proposed sample locations, please include the coordinates for the existing (historical) sample locations as well as the corners of the Inner Pier 1 Area.

b) The coordinates provided for the new sample locations are not consistent with the coordinates provided by on-line mapping services. Please correct.

c) Please clarify the description of the contingency sampling that will be implemented if no subsurface sediment samples can be collected from the three locations at the northern end of the Inner Pier 1 Area. For example, if none of the three subsurface sediment samples can be collected, will one subsurface sediment sample be collected from each of three of the remaining four sample locations so that three surface and three subsurface sediment samples will be collected in total?

Response:

a) Disagree. The requested information is not required for the SAP.

b) Further clarification required. The EPA does not provide any information regarding the problem or the referenced "on-line mapping services."

c) See response to General Comment 2.

August 20, 2009 Conference Call Resolution:

a) A table will be added to Figure 10-2 that includes coordinates for all proposed sample locations and applicable historical sample locations (i.e., P1, P1-48, P1-C1, and SBP-1). The coordinates for the corners of the Inner Pier 1 Area will also be labeled. All coordinates will be provided in the Connecticut State Plane NAD 83 system.

b) EPA used MapTech to evaluate the coordinates. After further review and the Navy's agreement to make changes to Figure 10-2 under a), the EPA accepted the proposed information and no further changes are required.

Specific Comment 5: p. 38, Worksheet #1

The bullet at the top refers to an EE/CA Addendum. EPA understood from the schedule that a revised Draft EE/CA, a Draft Final, and a Final versions will be prepared. Please clarify the scope of the revised EE/CA.

Response:

Agree with clarification. The Navy plans to prepare and issue draft and final versions of the Inner and Outer Pier 1 EE/CA. The Navy does not intend to issue a draft final version of the document. The bulleted item will be changed as follows:

- To develop an EE/CA, Action Memorandum, and Removal Action Work Plan for Inner and Outer Pier 1.

August 20, 2009 Conference Call Resolution:

The Navy is hopeful that a draft final version of the EE/CA will not be required. If regulator comments on the draft EE/CA warrant preparation of the draft final EE/CA, than the Navy will prepare the additional version. The Navy and EPA will resolve this issue after comments on the draft EE/CA are received. No further action is required for the UFP-SAP.

Specific Comment 6: Figure 10-2

a) Note that the demolished portion of Pier 1 extends farther north than depicted. Please update the figure to show the complete removal of the wooden pier.

b) The scaled distances between proposed sample locations as shown are significantly different from the actual distances based on the coordinates provided in Worksheet 14. Note also that the latitudes and longitudes provided in Worksheet 14 are different from those provided in Figure 2 of the Draft Final Remedial Action Work Plan for Pier 1 (August 2008). Please provide a scaled drawing so the placement of the sample locations is correct.

Response:

a) Agree. The figure will be revised to show the correct extent of the Pier 1 demolition. The demolition extended approximately 90 feet north of the line shown on Figure 10-2.

b) Disagree/Further Clarification Required. The scaled distances match the coordinates provided in Worksheet #14. The proposed sample locations provided in the UFP-SAP are not provided on Figure 2 of the Draft Final Remedial Action Work Plan. Therefore, the current figure appears to be correct. Further clarification from the EPA is required to address this comment.

August 20, 2009 Conference Call Resolution:

b) See Resolution of Specific Comment 4 b). No further changes are required.

Specific Comment 7: p. 39, Worksheet #15

The reference to 40 CFR §260.11 in the metals table should be corrected to 40 CFR §261.11. Please make this correction throughout the document.

Response:

Agree. The reference will be changed to 40 CFR §261.11.

Specific Comment 8: p. 39, Worksheet #15

Please discuss whether the data will be compared with the site-specific PRGs in addition to the criteria listed on Worksheet #15. If PRGs will be used, then a Worksheet #15B should be provided and populated with the Project Action Levels appropriate to those PRGs.

Response:

Disagree. The newly collected sediment samples will be analyzed for PCB-Aroclors and TCLP Metals. The data will be used to determine disposal requirements. The criteria provided in Worksheet #15 are appropriate for evaluation of the data. Comparison of the data to site-specific PRGs is not appropriate.

Specific Comment 9: p. 41, Worksheet #16

The deliverable due dates are not consistent with the other dates in this table. Please correct as appropriate.

Response:

Agree. The dates will be corrected to match the proposed dates in the Anticipated Date of Completion column.

Specific Comment 10: p. 43, Worksheet #17

Please clarify why the Navy is planning to collect surface sediment samples for TCLP analysis from the four locations that have previously been sampled. The highest metals concentrations, especially for lead, were generally at depth. Why shouldn't the TCLP samples come from the sediment most likely to have the greatest contamination?

Response:

The TCLP analyses are being performed to provide a preliminary indication regarding the likelihood of the sediment to be characteristic hazardous waste. Final determination of the nature of the sediment waste will be determined through ex-situ TCLP testing per the Remedial Action Work Plan.

Existing data for the four surface sediment samples (P1, P1-C1, P1-48, and SBP-1) selected to sample adjacent to had high PCB and lead concentrations. Lead concentrations were as high as 2,834 mg/kg in the samples. Therefore, these locations are appropriate locations to sample.

Specific Comment 11: p. 44, Worksheet #18

Collecting surface sediment samples for TCLP analysis does not appear to be appropriate given that metals concentrations are generally greater at depth.

Response:

Disagree. See the response provided for Specific Comment 10.

Specific Comment 12: Figure 11-1

This figure refers to an EE/CA Addendum. Please clarify the Navy's intention regarding the scope of the revised EE/CA.

Response:

Agree. The Navy will issue draft and final versions of the Inner and Outer Pier 1EE/CA. The figure will be revised accordingly.

August 20, 2009 Conference Call Resolution:

See the resolution for Specific Comment 5.

Specific Comment 13: Appendix E

Please include the minutes from the May 14, 2009 meeting in Boston that discuss the rationale for the TCLP sampling and also the minutes of the June 17, 2009 conference call.

Response:

Agree. The minutes from the May 14, 2009 meeting and June 17, 2009 conference call will be included in Appendix E.

Rich, Corey

From: Keckler.Kymberlee@epamail.epa.gov
Sent: Tuesday, September 01, 2009 4:21 PM
To: james.gravette@navy.mil; Rich, Corey; richard.conant@navy.mil; mark.lewis@po.state.ct.us; rtfinlayson@gfnet.com
Subject: Inner Pier 1 UFP-SAP

EPA reviewed the Responses to EPA's July 23, 2009 comments on the Draft Inner Pier 1 Sediment UFP-SAP (Initial Issue: August 12, 2009; Revised: August 31, 2009). The responses were sent by Corey Rich yesterday. The responses for the Inner Pier 1 SAP are acceptable. EPA has no further comments.

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